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Abstracts

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SESSION A1 EXISTING AND EMERGING SURFACE ENGINEERING PROCESSES

APPLICATION OF VIBROTERMOFLUID® METHOD FOR CYANIDING OF TOOL STEELS

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This paper describes the influence of fluidised bed treatment with vibration-aided active bed on both hot and cold-work tool steels. The applied technology was compared with typical fluidised bed treatment.

Heat treatment was performed on cold-work tool steel X160CrMoV121 (AISI D2) and hot-work tool steel X37CrMoV51.

Metallographic observations were carried out with the use of light microscope Olympus 1X70 and electron microscope Hitachi S-3500N equipped with energy dispersive x-ray spectrometer EDS, made by Thermo Noran company. The EDS was used to approximate the chemical composition of the surface layers, formed on the specimens. For metallographic examinations the microscope Axiovert 200 MAT from the Carl Zeiss company was also used, microstructures were stored in digital form with the use of AxioCam MRc5 camera. Friction wear tests were performed with the asd "3 rollers - cone" method, with 4 values of unit pressures: 50, 100, 200 and 400 MPa.

DIFFERENCES ON THE NITRIDED LAYER BETWEEN CLASSIC ACTIVE SCREEN PLASMA NITRIDING AND ACTIVE SCREEN PLASMA NITRIDING WITH A SEMISPHERICAL CATHODIC CAGE

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The Active Screen Plasma Nitriding (ASPN) is commonly used when regular surface hardening is necessary for product under treatment. ASPN technique presents more homogeneous surface coating than the Direct-Current Plasma Nitriding (DCPN) due to different process principles caused by interference of the metal screen working as cathode instead of the material under treatment. Cathodic cage in plasma nitriding refers to an active screen with a defined geometry in order to understand the physical parameters.

The purpose of this work was to study the differences between ASPN using a semispherical cathodic cage and the ASPN using a cylindrical regular cathodic cage. Through several procedures under similar parameters the tests were done with three conditions: with DCPN, with cylindrical cathodic cage in ASPN and with semispherical cathodic cage in ASPN. X-ray diffraction (XRD), scanning electron microscope analysis (SEM) and energy dispersive spectroscopy (EDS) were applied in the samples to characterize its nitrided layer. It was found with the XRD that the nitrided layer are not equal for each different condition applied to the process, with two iron nitrides found in different concentration in the sample surface. More Fe2-3N was found in the semispherical cathodic cage ASPN than was found in cylindrical cathodic cage ASPN and semispherical cathodic cage ASPN had no or very low concentration of Fe4N and cylindrical cathodic cage ASPN had one low intensity peak of Fe4N. The DCPN treated samples showed both nitrides with higher concentration of Fe4N than both ASPN treated samples. EDS analysis sustain that a nitrided layer was formed as it

does show presence of nitrogen along sample's profile. SEM analysis showed different surface morphology for each condition.

NANOMECHANICAL AND NANOTRIBOLOGICAL TESTING OF SURFACE ENGINEERED SYSTEMS AT ELEVATED (UP TO 750 ^{O}C) AND SUB-AMBIENT TEMPERATURES (DOWN TO - 30 ^{O}C).

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Surface engineering systems are used to provide engineers and material scientist with solutions to their problems. However the design and evaluation process can be extremely long in order to provide a final solution. In this current study we will present case studies to show the nanomechanical testing of surface engineering systems at both elevated[a] and sub-ambient[b] temperatures for various applications.

Surface engineering systems are developed so that they can be used in harsh and aggressive environments where the bulk material properties alone are not fit for purpose. Temperature is one of the many elements, which these systems need to be able to withstand. Testing is usually performed at room temperature, which clearly will produce performance data that cannot be completely relied upon. The nanomechanical testing techniques that are presented allow these systems to be tested at their in-service environmental temperature. This provides enhanced reliability of the knowledge of how such systems will behave in-service.

Results will be presented using nano-scratch, nano-impact and nanoindentation, the tested materials include DLC and Titanium along with SOFC materials.

The techniques presented will help to enable the surface engineers develop systems that are more suited to their application and can be developed and evaluated at a faster pace. The combination of the multidiscipline areas mathematical modelling and the relevant nanomechanical testing will provide vital tools to surface engineers of the future.
High temperature, Low temperature, Nanomechanics, Nanotribology

References:
a) J. Milhans, et al, Mechanical properties of solid oxide fuel cell glass-ceramic seal at high temperatures, Journal of Power Sources, 196 (2011) 5599
b) J.Chen, et al, Low Temperature Nano-Tribological Study on a Functionally Graded Tribological Coating Using Nanoscratch Tests, Tribol Lett, (2011)

PLASMA NITRIDING OF SPRAY-FORMED AL ALLOYS

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Thanks to their typical Si contents between 15...35 wt.-%, spray-formed Al alloys have a high level of wear resistance. However, additional surface protection treatments are taken into consideration in connection with their use for high-load applications, e.g. as power train components.

Within the last years considerable progress was achieved in the field of plasma nitriding of cast and wrought Al alloys. It is known that the nitriding behaviour depends on the elimination of the natural oxide layer and is essentially influenced by alloying elements. It is promoted by magnesium and impeded by higher contents of silicon.

First investigations on plasma nitriding of spray-formed Al alloys with a high Si content showed an excellent nitridability contrary to what was expected. It was possible to generate AlN layers with a thickness of up to 6 µm.

Despite the high Si content this positive effect is the result of the primarily solidified and homogeneously dispersed small (2 to 5 µm) Si precipitations.

The effect of the

- - alloying elements (Si, Mg, Fe and Cu content)
- - nitriding parameters (temperature, time, pressure)
- - surface roughness

on the nitriding behaviour, e.g. quality, thickness and properties of the layer, will be demonstrated using selected spray-formed Al alloys.

Different alloying contents of Al base materials were examined regarding their influence on both the nitride layer growth behaviour and the nitride layer properties (hardness, corrosion and wear resistance).

As known, nitriding leads to excellent antiscuffing capacity. But due to the abrupt change of surface hardness from a very hard AlN layer (14 GPa) to a soft Al matrix material (2.5 GPa), the loading capacity of the nitride layer is limited. For this reason the capabilities and limits of a subsequent heat treatment (age hardening) were examined.

LIQUID PHASE SURFACE ENGINEERING OF ALUMINIUM ALLOYS USING NOVEL ELECTRON BEAM DEFLECTION TECHNIQUES AND ITS INFLUENCE ON MICROSTRUCTURE - PROPERTY RELATIONSHIPS

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The application of lightweight materials such as aluminium alloys, especially in systems stressed due to tribological contacts, calls for an additional modification of functional surfaces according to the loading conditions. Not least because of a modern generation of beam deflection techniques available, electron beam (EB) liquid phase surface treatment is a possible and innovative solution for these requirements. The modification of microstructure and properties of these surface layers (0.4 ... 4.0 mm in thickness) is caused by rapid solidification and cooling by self-quenching, whereas layer properties (hardness, friction coefficient, wear rate) can be improved significantly, especially in combination with implemented additional elements (e.g. Cr, Co, Cu, Fe, Ni) and/or hard particles (WC) during local EB processing (surface alloying/dispersing). Experimental results show 2 to 7 times higher hardness and scratch energy density values as well as 10 to 25 times lower wear rates and additionally decreased friction coefficients in comparison to the untreated base materials.

The paper deals with the current investigation results of EB surface alloying and dispersing of commercial cast, wrought and spray-formed Al alloys, using high frequency beam deflection. The results will be discussed according to the interactions between the EB and the material and its effects on the layer microstructure and the characteristic layer properties. Furthermore, detailed researches into electrochemical corrosion and friction/wear behaviour and their damage characteristics are subjects of discussion.

The EB technologies discussed offer new possibilities for improving the service life and reliability of engine components, among others.

THE STUDY ON THE PHASE TRANSFORMATION OF AISI D2 TOOL STEEL AFTER HIGH TEMPERATURE GAS NITRIDING AND TEMPERING.

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Recently, High temperature gas nitriding (HTGN) has been introduced as a method for surface modification. The HTGN treatment involves a diffusion process for nitrogen to permeate the surface of steel through heat treatment in N_2 gas atmosphere at high temperatures. Despite of many studies on the HTGN treatment, there has been no report on the HTGN treatment for tool steel. In this study, the effects of the high temperature gas nitriding (HTGN), tempering and subzero treatment of the AISI D2 steel have been investigated. The HTGN treatment was carried out at 1050℃, 1100℃ and 1150℃ for 1hr. in an atmosphere of 1kg/cm^2 nitrogen gas. Tempering and double-tempering were performed at 550℃ for 1hr. The surface layer of HTGN- treated steel appeared the precipitates of M_2N, M_7C_3 and $M_{23}C_6$ in the matrix of austenite. However, the interior region, where the nitrogen was not permeated, exhibited martensite with the precipitation of carbides. The nitrogen content of the surface layer appeared ~1.35 wt.%, ~0.83 wt.% and ~0.56 wt.% at the HTGN treatment temperature of 1050℃, 1100℃ and 1150℃, respectively. The maximum value of surface hardness of steels after double-tempered and subzero-treated are 828Hv, 960Hv, 750HV with HTGN treatment at the 1050℃, 1100℃ and 1150℃, respectively. It increased above 230~420Hv compared with the HTGN treated steel due to the transformation from retained austenite to martensite and existence of fine precipitates.

SESSION A2 MODELLING OF PROCESSES AND PHENOMENA

A CFD APPROACH FOR DESIGNING LOW PRESSURE CARBONITRIDING

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The low pressure carbonitriding of steel is a process under development which aims at case hardening of steel parts that are exposed to high stresses, such as gear box components, shafts and high pressure pumps. Those components need a hard and wear resistant surface while retaining a ductile core to resist cyclic stress loads.

The process is carried out in vacuum furnaces at pressures below 50 mbar and at temperatures ranging from 750 °C to 900 °C. In consecutive steps the steel parts are exposed to a carburizing gas (common carbon donors are propane (C_3H_8) and acetylene (C_2H_2)) and to a nitriding gas (usually ammonia (NH_3)). The order, the length and the number of carburizing/nitriding periods are variable and depend on the required physical properties of the case hardened steel parts.

In an ideal process, carbon and nitrogen are taken up by the steel according to the following two reactions:

C_2H_2 -->2 C_{steel} + H_2 and 2 NH_3 --> 2 N_{steel} + 3 H_2

However, in reality unwanted side reactions take place that lower the availability of carbon and nitrogen in the gas phase and contaminate the heat treatment equipment, increasing the maintenance rate of the furnace. Acetylene, for example, reacts in addition type reactions to vinylacetylene (C_4H_4) and benzene (C_6H_6), common predecessors of tar and soot. These foul the equipment and dissolve in the oil of the vacuum pumps causing run time reduction and wear. The nitrogen donor ammonia reacts to hydrogen and molecular nitrogen; molecular nitrogen can be considered inert under carbonitriding conditions and therefore less nitrogen is available for uptake by the steel parts.

To optimize the low pressure carbonitriding process, it is of great importance to understand the homogeneous gas phase reactions and the processes on the steel surface in combination with the diffusion into the surface layers of the steel part. At the Engler-Bunte-Institut, in close cooperation with industry, CFD models for the carburizing reactions, the carbon uptake and for the carbon diffusion have been developed and verified in an industrial sized furnace. This paper presents the latest improvements on the nitriding model, which ultimately leads to a CFD model that is capable of predicting the carbonitriding process, including the transient gas phase composition, as well as the carbon and nitrogen concentration in the steel sample.

NUMERICAL SIMULATION OF THE GROWTH DYNAMICS OF MONOPHASE LAYERS IN THE PROCESS OF NITRATION.

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The article deals with the physical aspects of phase formation during nitridation of iron. It is noted that the problem of nitrogen diffusion in the crystal lattice of iron can be attributed to problems of "Stephan" type with a moving boundary, which is complicated by the presence of two phases, which change their position depending on the environmental conditions of exposure - the temperature of the saturating atmosphere of nitrogen and its potential. On the contrary of the classical approach based on partial differential equations, the paper proposes to use the integrated diffusion equation for the numerical solution of such problems.

Deduced integral equations contain both the initial and boundary conditions of the problem and the conditions at phase interfaces, which allows the use of non-adaptive mesh subdivision. Integral formulation of the problem also allows us to estimate the error produced an approximate solution and to obtain a stable scheme for the numerical solution (including the two-and three-dimensional case). There is a program complex, with which it is possible to control the technical process of nitridation by solving the problem of diffusion of nitrogen with a changing external conditions.

MODELING THE NITRIDING OF QUENCHED AND TEMPERED STEELS

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A new model is being developed to predict the Nitrogen and hardness profiles in the nitrided case of quenched and tempered alloy steels. The model utilizes modified Leher Diagrams for the nitrided alloy as well as isopleths for the alloy. The kinetic calculations include diffusion

with a precipitation reaction as well as experimental results. The results for AISI 4140 steel will be presented and discussed.

ESTIMATION OF THERMAL BOUNDARY CONDITIONS OCCURING DURING QUENCHING BY GRADIENT BASED AND GENETIC ALGORYTHMS

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The estimation of thermal boundary conditions obtained during heat treatment processes is an essential requirement to characterize the heat transfer. In this work numerical methods based on gradient optimization techniques and genetic algorithms used for the esimation of heat flux and heat transfer coefficient are analized and compared for prediction of HTC. The comparision study of the techinques applied are based on computational results performed by heat treatment simulations.

AN ACCURATE PREDICTION OF TEMPERATURE IN INDUCTION HEATING WITH CONSIDERATION OF TEMPERATURE-DEPENDENT BH CURVES

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Induction heating is a popular heating method in industrial heat treatment. Optimum heating conditions of induction heating (e.g., heating coil dimensions, coil current, clearance between workpiece and coil) are determined by trial and error, and that usually takes long time. In order to determine heating conditions in a short time, simulations for temperature prediction are often employed recently.

In induction heating, a metallic material is heated from the surface by eddy currents. The amount of eddy current is determined by the BH curves of the material. Therefore, it is quite important to consider temperature-dependent BH curves for accurate simulations.
An error between simulation and actual temperature need to be less than ±25K for determination of heating conditions. However, in conventional simulations, it has not been achieved because of difficulty in the measurement of BH curves at high temperatures.
In the measurement of BH curves, a ring specimen with two winded enamel-coated copper wires for magnetic field excitation and magnetic flux detection is usually applied. But it is not possible to measure BH curves over 473K using above-mentioned apparatus, due to insufficient heat resistance of electrical insulation property of the enamel coating.
For the purpose to improve accuracy of simulations, a new measurement method of temperature dependence for BH curves has been developed. In order to overcome poor electrical insulation property of the enamel coating, the new wires which consists of silver wire as inner conductor and ceramic-braided tube as electrical insulant has been made and applied to the measurement. A direct-current self-registering fluxmeter was employed for measurements. The range of applied magnetic field was ±8000 A/m.

Using the developed wire, BH curves of medium carbon steel (AISI 1045) have been successfully measured up to 1073K. From room temperature to 473K, maximum magnetic flux density is decreased approximately 10% due to paramagnetization of cementite in specimen. From 973K to 1073K, drastic decreasing of maximum magnetic flux density due to phase transformation from ferrite (ferromagnetic) to austenite (paramagnetic), and disappearance of hysteresis around the Curie point can be observed.

In order to confirm accuracy of simulation, experimental and calculation results were compared.

Induction heating simulation is conducted by coupled analysis of electromagnetic and thermal FEM calculations. In the electromagnetic calculation, the measured BH curves are considered up to 1073K. Through the entire heating process with heating rate of 400K/sec, the result of simulation corresponds to experimental results within an error of ±20K.

MODELLING THE RE-AUSTENITISATION OF STEELS

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A model has been developed for the reaustenitisation of steels that utilises a modified Johnson-Mehl-Avrami equation. The governing principles of the model are as follows:

That the kinetics of re-austenitisation is limited by C diffusion. For the case of a starting microstructure of ferrite + pearlite, transformation occurs by pearlite first transforming to austenite of the same composition as pearlite, giving rise to a transformed microstructure of ferrite + austenite. When starting with a tempered ferrite + carbide microstructure, the transformation will be similar in that some of the ferrite will transform with the carbide, such that microstructure on transformation will be ferrite + austenite. This mixture of ferrite + austenite transforms to austenite on further heating. On completion of reaustenitisation, the C concentration of austenite is taken to be the same as that of the original alloy.

A potentially quite different transformation may occur at very fast heating rates because it is thermodynamically possible for ferrite, both in the pearlite and the original ferrite grains, to transform directly to austenite in a massive, diffusionless way. Such a transformation is also considered in the present approach. Calculation of Time Temperature Austenisation diagrams is made and various examples will be shown. Calculation of temperatures for homogenous austenite to form as a function of heating rate are also made as is the resulting grain size.

SESSION B1 EXISTING AND EMERGING SURFACE ENGINEERING PROCESSES

METALLURGICAL ANALYSIS AND COST CONSIDERATIONS OF TIALN COATINGS ON HOT WORKING DIES

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Failure mechanisms of titanium aluminium nitride coatings on hot extrusion and forging dies have been studied in an attempt to evaluate the performance of these hard layers during hot working operations. The study was performed by failure analysis of typical forming dies and examination of test specimens coated under the same condition by cathodic arc evaporation on H13 hot work tool steel. The coatings and interface regions were characterized by scanning electron microscopy, AFM and x-ray diffraction. Microhardness and nano-indentation tests were conducted to evaluate the mechanical properties of surface layers.

Frictional behaviour of the coated specimens was studied by a pin-on-disc wear tester. It was found that TiAlN coating had a rather low coefficient of friction at room temperature starting from about 0.2 and increased gradually as the counterface was abraded. SEM examination of the wear tracks and microanalysis revealed formation of aluminium oxide as a dense, adherent film. Increase in lifetime at the most severe condition has been 70% but up to three times in other cases. TiAlN coatings, compared with the more popular TiN, are about 50%

more expensive but they are cost effective in cutting and forming tools. In forging dies, coating cracks were observed at the edges whereas in hot extrusion, uniform cracking occurred before coating spallation. Experimental results suggest that high aluminium TiAlN acts as a reservoir for aluminium and forms protective Al_2O_3 on the surface when heated. Reducing adhesion to workpiece material and sufficient toughness to resist brittle fracture are key factors for good performance of TiAlN.

FORMATION OF PHOTOCATALYTIC FILM ON TITANIUM SUBSTRATE USING AN ELECTROLYTIC JET

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The anatase type TiO_2 (A-TiO_2) shows superior photocatalysis even under the weak light. It is possible to decompose toxic substance with the difficult decomposition (NO_X, C_2HCl_3 etc.) of ppm or ppb level. Since the valence band of A-TiO_2 is lower than that of rutile type TiO_2 (R-TiO_2) by 0.1eV, the A-TiO_2 is more oxidizable. A-TiO_2 is irreversibly transformed into R-TiO_2 in the high temperature. Anodization can form a TiO_2 film at high voltage over about 100V and high current density. Since high current density is necessary, the film formation to large area is difficult. It is disadvantageous that it can not produce a A-TiO_2 film except for Ti substrate.

However, the fabrication of a pure A-TiO_2 film is possible, since the binder is not used. The electro-plating was carried out, in which an electrolyte jet was applied to improve the deposition rate. The resultant deposition rate of plating with the electrolyte jet is approximately 15 times faster than that of conventional electro-plating. In this study, research results in the jet electro-plating is applied to A-TiO_2 film formation to large area Ti substrate.

CVD HARDIDE COATINGS PROTECT INTERNAL SURFACES AND COMPLEX SHAPE PARTS AGAINST WEAR AND EROSION.

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Hardide™ is a new family of nano-structured CVD Tungsten Carbide/Tungsten coatings used to increase the life of critical parts operating in abrasive, erosive and chemically aggressive environments.

The Hardide coatings allow optimise design for improved operation of critical parts of pumps, valves, oil drilling tools. In most applications the economics of the coating is defined by the reduced down-time and enhanced reliability of expensive equipment, such as an oil directional drilling tool.

The Hardide-T coating consists of nano-particles of Tungsten Carbide dispersed in a metal Tungsten matrix. This structure gives it a unique combination of enhanced hardness (1100-1600 Hv) with excellent toughness, crack and impact resistance. An advanced CVD coating technology enables control of the hardness and toughness of the coating, and thus to achieve optimum protection against wear and erosion. The coating hardness inhibits the micro-cutting mechanisms of wear and erosion while its toughness, ductility, residual compressive stresses and homogeneous micro-structure prevent fatigue micro-cracking/chipping and platelet mechanisms of erosion.

The coating is typically 50 microns thick - exceptionally thick for CVD - and withstands 3000 microstrain deformations without any damage; this deformation will crack or chip most other thick hard coatings. The gas-phase CVD process enables the uniform coating of internal surfaces and complex shapes such as pump cylinders, impellers, extrusion dies or ball valves. The need for post-coat grinding is eliminated in the majority of applications thereby reducing cost and turnaround time compared to traditional coating methods.

The pore-free Hardide coating protects parts from corrosion, acids and aggressive media. It is an attractive replacement for Hard Chrome, which is being phased out due to environmental legislation, especially for coating complex shapes and internal surfaces.
The coating can be applied on various grades of steel, stainless steel, Ni, Cu, Co alloys, Titanium.

In a number of tests Hardide CVD coatings were compared with other traditional hard facing technologies, such as HVOF WC/Co coating, Hard Chrome plating and PVD coatings. Each of these surface engineering technologies has its specific strengths and limitations, which determine the most suitable applications. The CVD Hardide coatings are particularly suitable for complex shape parts with precise dimensions, or where internal surfaces need protection against wear and erosion. These applications are often difficult to coat by other technologies, thus the Hardide CVD coating is often complementing other coatings rather than replacing them, and helps expand the range of surface engineering applications.

THE INVESTIGATION OF CR-AL-SI-N FILMS DEPOSITED ON CEMENTED CARBIDE SUBSTRATES FOR HIGH-SPEED DRY CUTTERS

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Nanocomposite Al-Cr-N and Cr-Al-Si-N films were deposited on cemented carbide substrates by multi-arc ion plating (MAIP). The thermal stability of Al-Cr-N and Cr-Al-Si-N films was investigated by annealing within a range of temperatures 700-1100 ℃ during 2 hours and DSC at argon atmosphere in vacuum. The microstructure and mechanical properties of Cr-Al-Si-N films were studied by using X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), high resolution transmission electron microscope (HRTEM), scratch test, nano-indentation and friction-wear tests. The results showed c-(Cr, Al)N annealed at 900 ℃ for 2 hours in vacuum at 5×10^{-3} Pa were decomposed into h- Cr_2N and c-AlN. The addition of 3.5 at.% Si in the Cr-Al-N film dramatically improved the mechanical properties and thermal stability of the films up to 1100 ℃. The mean hardness value of the Cr-Al-$Si_{3.5\ at.\%}$-N film sharply increased to 42 GPa because of the formation of Si_3N_4 phase and decrease in grain size, besides on internal compressive stress during deposition. After annealing with more than 900 ℃, the hardness of Al-Cr-N and Cr-Al-Si-N films were sharply increased by 30~40% because of the formation of hcp-Cr_2N phase. Compared to Al-Cr-N films, the friction coefficient and the wear rate of Cr-Al-Si-N films decreased obviously. And the wear mechanism of Cr-Al-Si-N films was analyzed by SEM-EDS and XPS. As such, the Cr-Al-Si-N film with 3.5 at.% Si by MAIP has a good potential in the application for high-speed dry cutters. The fundamental mechanism will be discussed in terms of the thermal stability of the Cr-Al-Si-N nanocomposite films.

THERMAL FATIGUE OF HOT WORKING STEEL AFTER HYBRID SURFACE TREATMENT

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The application of surface treatment methods like ion nitriding, PVD coatings and their combination in hybrid treatment have been effective to reduce oxidation, corrosion, erosion and wear. However, it is still uncertain whether nitriding and hybrid treatment have any real effects on the decrease of the nucleation and growth of thermal fatigue cracks on the surface. This paper presents the results of thermal fatigue investigations of nitrided layer and different composite layers "nitrided layer / PVD coating" (TiN, CrN, TiAlN) obtained on the X40CrMoV5.1 hot working steel. The ion nitrided only and three different hybrid-treated substrates were investigated based on the intensity of thermal fatigue cracks observed after testing. The investigated nitrided layer and composite layers were obtained with the use of the hybrid surface treatment technology, which consist of ion nitriding followed by arc-evaporation coating deposition. An apparatus based on high frequency induction heating and water spray cooling was used for thermal fatigue tests under the following conditions: maximum temperature 600°C, minimum temperature 80°C and three different amounts of thermal cycles: 500, 1000 and 2000. The thermal fatigue intensity of the nitrided layer and three different composite layers "nitrided layer / PVD coating" were measured according to surface crack density and crack propagation depth after different amount of thermal cycles. Finally, based on the obtained results, the influence of different PVD coatings in composite layer "nitrided layer / PVD coating" on the increase of thermal fatigue resistant of hot working steel was discussed.

SESSION B2 MODELLING OF PROCESSES AND PHENOMENA

THE NUMERICAL SOLUTION OF THE PROBLEM OF PHASE TRANSITIONSON ON LARGE-SCALE NON-ADAPTIVE GRIDS.

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The use of numerical methods based on finite difference method (FDM) and finite element method (FEM), often leads to oscillations of the approximate solutions in the contact area of media with different thermophysical properties. As a result, the accuracy of the calculated data to estimate the error is reduced and the resulting approximate solution is not possible. In addition, the increased complexity of the problem of phase transition is the problem of determining the boundaries of the front of melting (crystallization), which leads to the need to rebuild the finite-difference or finite-element mesh at each time step (solution on adaptive grids).

We consider the approach where a uniform heat conduction problem is described in the entire region of the solution, and the resulting integral equation for heat flow to automatically take into account the conditions at the discontinuity of the coefficients of thermal conductivity and heat capacity. In this case the numerical solution of moving boundary can be performed on partitions of the grid, independent of time and position of the interface at any time determine not only at the nodes, but also in the inner parts of the elementary segments, which are divided region solution .

NUMERICAL SIMULATION OF CARBURIZING AND SUBSEQUENT HEAT TREATMENT OF STEELS

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Numerical modelling of manufacturing processes by means of finite element method or by other mathematical techniques is one of the tools which are used routinely in many branches of industry. Purposeful utilization of numerical models typically leads to significant

improvement in the effectiveness of new process design and development. In the field of heat treatment, however, computer modelling is used scarcely. It is used almost exclusively for predicting stresses arising from heating and cooling sizable tools and parts. There is very little experience in using computer simulation in thermochemical treatment of tools and machine parts still.

The present paper presents a numerical model of diffusion-based enrichment of surface of steel gear wheels with carbon during carburizing at various carbon potential levels. The model was constructed at the company COMTES FHT for DEFORM HT, a software tool based on the finite element method. Computer simulation allows the carbon concentration depth profile to be predicted with high accuracy for various parameters of the carburizing process. It can also describe the resulting distribution of phases, hardness profile and distortion upon quenching in various quenching media.
The model was verified by testing on machine parts in collaboration with the company WIKOW GEAR, a world's leading manufacturer of large industrial-grade gear boxes. Several 370 mm diameter gear wheels from 18CrNiMo7-6 steel were carburized, quenched and tempered. The comparison between data measured on the wheels and results of the numerical model showed that the prediction of phase composition, surface hardness and resulting distortion of wheels was very accurate.

PREDICTION OF MECHANICAL PROPERTIES OF QUENCHED AND TEMPERED STEEL AND CAST STEEL

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The influence of processing parameters of steel casting, hot working and heat treatment on mechanical properties of quenched and tempered steel and cast steel was investigated. Representatives of mechanical properties were yield strength and Charpy-V notch toughness. Yield strength was measure of steel strength, and Charpy-V notch toughness was measure of steel toughness. Experimental procedure was done using the 2^{7-4} factor experiment on steel EN 42CrMo4 and cast steel EN GS-42CrMo4. Using this factor experiment influence of temperature of casting, cooling rate during the casting, as well as, application of hot working, normalization, homogenization on yield strength and Charpy-V notch toughness was done. It was found out that yield strength is deterministic by based processing parameters of casting, as well as, of quenching and tempering, but it is insensitive on investigated process parameters. Charpy-V notch toughness is sensitive on application of hot working, temperature of casting, and on interactive effect of cooling rate during the casting and application of hot working.

Test results of investigated mechanical properties were verified by microstructure analysis. It was found out that hot worked and normalized specimens have refined microstructure. Austenitic grain size of investigated steel treated by hot working has ASTM number of previous austenite grain size equal to N.6-N.7, while austenitic grain size of cast steel has ASTM number of previous austenite grain size equal to N.4-N.5. Steel treated by homogenization and hot working has finer non-metallic inclusions. Using the ASTM E 45 classification it was found out that by application of homogenization and hot working the inclusion number was drop down from 4-4½ to 1½-2.
Mathematical expressions for prediction of yield strength and Charpy-V notch toughness of steel and cast steel were established. Established mathematical expressions were applied in computer simulation of mechanical properties of steel and cast steel workpiece of complex form. Mechanical properties of quenched and tempered steel and cast steel can be successfully calculated by the proposed method.

CALCULATION OF THE TEMPERATURE DEPENDENCE OF MAGNETIC PERMEABILITY FOR STRUCTURAL STEELS

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The temperature dependence of magnetic permeability μ [T] is a critical parameter required for the calculation of the optimum frequency leading to a given penetration depth in induction heating.

μ[T] depends on both the intrinsic variation with temperature of μ for the ferritic component of the microstructure, and on the temperature dependence of the volume fraction of that phase V_F[T]. Only room temperature data is available in many cases; hence there is a need for better predictive modelling, especially for steels, where the temperature dependence is markedly non-linear. This paper discusses the methodology by which such a predictive capability has been achieved and its validation against experimental data.

The value of μ [T] is taken as proportional to the saturation magnetisation and inversely proportional to the Coercive force H_c [T] which, in turn, is related to the hardness of the alloy. It is shown that, for structural steels, a simple law of mixtures can then be applied to combinations of austenite and ferrite-carbide aggregates, assuming μ =1 for austenite and carbides. The JMatPro software being used has the proven capacity to calculate the fraction of ferrite at various heating rates (see parallel paper in this conference) and can also provide the variation of Curie temperature versus composition, from a working value of the saturation magnetisation can be derived.

This means that the only required user inputs are the hardness and composition of the alloy in question. Values of μ [T] for structural steels of any desired composition can therefore be calculated, together with the potential variations due to compositional changes within specification.

ENHANCED REQUIREMENTS ON SURFACE QUALITY OF OUTER CAR BODY SHELLS IN AUTOMOTIVE LIGHTWEIGHT DESIGN

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Demanding automotive design in the interaction of modern lightweight strategies to reduce the car body weight as well as legislative regulations impose higher requirements on future car body developments. This trend leads to thinner sheet metal blanks and indicates higher requirements on narrow process windows in the entire manufacturing process to ensure the surface quality of outer shell panels. Especially thermal loads within the coating process might cause local shape deviation. Further developments in multi-material-design for car body components induce material configurations with a complex deformation behaviour due to the different material characteristics of thermal expansion in thermal processes. For these reasons there is a need to improve the prediction of the surface quality in the early car development process using numerical simulation methods. The influence of process paramters according to the surface quality is shown and integrated into the process simulation.

SESSION C2 IFHTSE PROJECT: GLOBAL 21

SURFACE ENGINEERING : LIMITS IN ENGINEERING APPLICATIONS

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Recently the 6th China International Conference on Surface Engineering in Xi'an brought once again in focus the need to define what surface engineering is in terms of mechanical engineering. The fast development in materials science and the increasing need for surface modification to meet the most critical applications in mechanical systems but also in bioengineering or other areas raised questions how far the "classical" definition of surface engineering has to be adapted to the new challenges.

The paper outlines the extremely wide area of surface engineering covering applications from liquids to thermal barrier coatings and bio engineering. It tries to formulate a possible definition and acceptable limits for surface engineering in interpretation of the International Federation for Heat Treatment and Surface Engineering.

As a first approach those processes are considered which involve heat or thermal and chemical effect for modifying the surface properties of the materials. The problem of the interface is analized as the affected thin surface area of the substrate should be critical for the related processes. Typical examples are quoted including also borderline special cases which need interpretation (concerning especially nanotechnology and its "derivates")
The paper is part of the "Global 21" project of IFHTSE and is intended to trigger a debate on this important subject influencing also the future of the Federation.

TECHNOLOGY AND KNOWLEDGE TRANSFER IN SURFACE ENGINEERING, SUPPORTED BY INTERNATIONAL PROGRAMMES

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Higher Education plays an enormously important role in technology and knowledge transfer, key factors in economic competitiveness. Its efficiency is even more in the centre of interest because of the recent economic crisis, but on the other hand, only restricted resources are available for the modernisation of HE. Responsiveness for dynamically changing and widening training needs is evidently considered by academics, but their focus is limited mainly to the content. Less attention is paid on the methodology of teaching: engineers are taught by engineers, applying the same methodology as they themselves have been taught - while extended content and complexity should require more effective, modern pedagogical approaches and tools. Academics have to recognise: we cannot "transfer" all the knowledge, what our students will need in their life-time as engineers - instead, we should provide them solid fundamental knowledge on the basics, and we should equip them with skills of knowledge generation, finding and critically evaluating, filtering the enormous mass of information, integrating them into their own problem-solving competencies. HE should also reconsider its role in continuous education.

Parallel to the Global 21 project - which also analysed the education aspects of HT&SE, two EU supported projects developed new methodology and training materials in the topics of Surface Engineering. 'INNOVATE' Project - International On-Line Voc@tional Training in Surface Engineering, 2001-2004, Leonardo Programme - aimed at focusing the methodology: based on a flexible, resource-based curriculum design system, a set of multilingual training materials have been developed for testing the interface of teaching material / electronic delivery systems. Built on these experiences, the MinSE project - EU Socrates programme, 2006-2009 - aimed at designing, developing and testing a Bologna-conform course leading to a European Master's qualification in heat treatment and surface engineering. The part-online, part face-to-face course has been developed by a consortium of five universities and five industrial partners, while IFHTSE provided professional networking background.
This paper will shortly summarise the added values and practical experiences gained by the international collaboration in joint course development at the University of Miskolc. Moreover, it will outline a vision of expanding such joint efforts for developing even more effective,

modern pedagogic methods, ICT tools and open educational resources for improving the effectiveness of technology and knowledge transfer in Surface Engineering.

SESSION D1 FERROUS PROCESSES AND PHENOMENA

EFFECTS OF SHOT-PEENING, ARTIFICIAL CORROSION PIT AND DIFFUSIBLE HYDROGEN ON THE FATIGUE PROPERTY OF HIGH STRENGTH SPRING STEEL

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In order to improve the strength of suspension springs, it is necessary to improve corrosion fatigue strength of suspension spring steels. However, the dominant factor of corrosion fatigue property of suspension spring steels is not clarified enough. Therefore, in order to investigate the dominant factor of corrosion fatigue property of high strength suspension spring steels, fatigue test and delayed fracture test were carried out for quenched and tempered SAE9254 with the tensile strength of 1800MPa. The effects of toughness, compressive residual stress by shot-peening, artificial corrosion pit depth and diffusible hydrogen content on the fatigue strength were studied by rotation fatigue test. The toughness was controlled by changing prior-austenite grain size. In the case of specimen having straight part and specimen having an artificial corrosion pit which is smaller than compressive residual stress depth, the 10^7 cycle fatigue limit is improved by shot-peening, and the fatigue limit and fatigue strength for finite life decrease gradually with increasing diffusible hydrogen content. On the other hand, in the case of specimen having an artificial corrosion pit which is deeper than compressive residual stress depth, the fatigue limit remarkably decreases and the effect of diffusible hydrogen on the fatigue limit is almost negligible. The effect of the toughness on the fatigue limit and delayed fracture property is relatively small.

EXAMINATION OF DUCTILE FRACTURE MECHANISM FOR 0.2%C STEELS BY VOID OBSERVATION

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It is important to elucidate the mechanism behind ductile fracture to analyze the ductile fracture surface. In order to elucidate the ductile fracture mechanism, load-displacement curves were firstly obtained from 0.2%C carbon steels, the tempering temperatures of which changed from 300℃ to 500℃ after quenching. V-notched tensile specimens as well as Notch-less tensile specimens were employed to focus attention on the region of stress concentration. Energy to failure was calculated from load-displacement curves obtained from each tensile test. It was revealed that total energy to ductile failure did not depend on the tempering temperature of both notch-less and V-notched tensile specimens. Focusing on local deformation, local deformation energy did not depend on the tempering temperature of the notch-less tensile specimens, while it depends on the tempering temperature of V-notched tensile tests. The energy increases as tempering temperature. Both dimples of ductile fracture surface after notch less and V notched tensile tests are shallower and larger as tempering temperature. Because ductile fracture surface of notch-less tensile specimen was similar to that of V-notched specimen in the same tempering temperature, the effect of tri-axial stress by the notch was considered to be small. In addition, distribution of the voids and the distance from the fracture histogram after ductile fracture was examined by using the image of AsB (Angle Selective Backscattered Electron Detector) by LV-SEM. AsB images exhibit topographic contrast. It was revealed that sum of void area and number of voids were decrease with increasing distance from fracture surface. The circularity of voids was higher for small voids, lower for big ones. When voids nucleated, the circularity of voids was high. As

voids growth, the circularity of voids became low. With a notch, voids nucleated area was closer to the fracture surface than notch-less. These findings showed that local deformation energy except localized necking by V-notch were increased as tempering temperature. V-notch made voids nucleation centered near ductile fracture surfaces. The histogram showed number of voids and area of voids were larger near the fracture surface, the circularity of voids were low.

THE BRITTLE-TO-DUCTILE TRANSITION IN HIGH-NITROGEN AUSTENITIC STAINLESS STEELS

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Nitrogen is one of the austenite formers which can be used instead of nickel. Solute nitrogen increases yield stress with a large work hardening rate while it induces the embrittlement at low temperatures. The lost of ductility at low temperatures is a fatal for their utilisation as structural materials. In ferritic steels, the low temperature embrittlement is caused by the strong temperature dependence of dislocation motion due to the high Peierls barrier against the dislocation glide while austenitic steels generally do not exhibit low temperature embrittlement due to their low Peierls barrier. So it is necessary to elucidate the mechanism behind the low temperature embrittlement in high-nitrogen nickel-free austenitic stainless steels.In the present study, the brittle-to-ductile transition (BDT) behaviour of high-nitrogen austenitic stainless steel was investigated.

1.1 mass% nitrogen was added into Fe-25mass%Cr by using solution nitriding process at 1473K for 72ks in nitrogen gas under the pressure of 0.1MPa. The specimen was water-cooled not to cause austenite decomposition and induce nitride precipitates on the cooling. The solution nitrided steel was subjected to isothermal heat treatment in dual phase region at 1173K for 0.3ks. Finally, the isothermally heat-treated steel was re-austenised at 1473K. Four-point bend tests were performed, changing the testing temperature and the crosshead speed.

The samples fractured without apparent deformation at low temperature while they plastically deformed at elevated temperatures. The BDT temperature was defined as the temperature at which exhibits the highest apparent fracture toughness. The BDT temperature increases as the crosshead speed. The deformation rate dependence of the BDT temperature is subject to the Arrhenius type equation. The value of the activation energy was found to be 1.6eV, which suggests that dislocations hardy move at lo temperatures. The obtained value of the activation energy exhibits much higher than that from low-carbon steels. It is speculated that the difference of the value of activation energy is due to the difference of the controlling mechanism behind dislocation motions between in bcc and fcc structures.

MARTENSITIC TRANSFORMATION IN A FE-NI-0.8WT%C ALLOY AT SUB-ZERO CELSIUS TEMPERATURES

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Martensitic transformation in a model Fe-Ni-0.8wt%C alloy was investigated at sub-zero Celsius temperature.

Samples were austenitized and quenched, whereafter isochronal (constant cooling rate) and isothermal sub-zero Celsius treatments were applied. Dilatometry and (vibrating sample) magnetometry were used for describing the overall kinetics of the transformation in terms of the Johnson-Mehl-Avrami- Kolmogorov kinetics. The degree of transformation was also investigated with in-situ synchrotron X-ray diffraction by probing austenite and martensite

Bragg reflections. The state of internal stress and strain in the constituent phases was investigated at room temperature with (synchrotron) X-Ray diffraction, both prior to and after sub-zero Celsius treatment. Electron microscopy was applied to investigate the morphological changes upon martensitic transformation.

EXAMINATION OF PLASTIC DEFORMATION BEHAVIOR FOR DUAL-PHASE STEELS BY NANOINDENTATION TEST

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Mechanical properties such as strength and elongation of dual-phase steels are influenced by the properties of each phase and the difference of hardness, which affects stress concentration and void formation in grain boundaries. In this study, the behavior of plastic deformation in each phase of dual-phase steels was firstly investigated by using nanoindentation tests. Next, the plastic deformation process on two dual-phase steels; ferrite-bainite and ferrite-austenite, where the hardness ratios of two phases are different were examined by FEM analysis and the voids observation.

The cross-section surfaces of the indented regions in ferrite and austenite steel were observed by using FE-SEM. The indent depths in both ferrite and austenite were much smaller than those of the grain size. The hardness profile including grain boundaries of different phases were examined.

In general, the hardness ratio (HR) of ferrite to bainite was larger than that of ferrite to austenite. It is estimated that the voids formation behavior in the grain boundaries of dual-phase steels depends on the HR of each phase. In this study, the voids of specimens after tensile tests were observed by FE-SEM. In the nanoindentation hardness profiles, the HR of ferrite-bainite steel was 1.54, while the HR of ferrite-austenite steel was 1.32. The local strain distribution was obtained by FEM analysis which was conducted by using the nanoindentation hardness for one of the material components. The FEM analysis showed that the local strain of ferrite-bainite steel under the tensile stress of 20 % was concentrated in the interface of ferrite-bainite. On the other hand, the strain of ferrite-austenite steel under the tensile stress of 25% was concentrated in the soft phase of ferrite. As a result of the voids observation, the amount of voids in ferrite-bainite grain boundary was larger than that in ferrite-austenite one. These findings show that void nucleation tend to be occurred in the local strain concentrated area.

The main results obtained in this study are as follows:
(1) The hardness of each phase in dual phase steels can be measured exactly by nanoindentation test.
(2) The voids formation process in grain boundaries of dual phase steels can be estimated by FEM analysis and voids observation of FE-SEM.

BASIC TENDENCIES IN PROGRESS OF THEORY IN PRACTICE OF THERMO-CHEMICAL TREATMENT OF STEELS

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A surface of metal is of primary importance in providing of mechanical and some of physicochemical properties of machine parts as it is exposed to high loadings: ware, fatigue stress, corrosion, etc. That's why today we see clear trends to intensive development of

technologies of surface strengthening of metallic materials. Forming of strengthened surface layers on metals is achieved by purposeful realization of given structural state by different methods of actions. For surface modifying processes of thermo-chemical treatment are widely used. Among such methods nitriding is the most simple and effective technology. The analyses of Russian and foreign researches in the field of thermo-chemical treatment, and, in particularly of nitriding has shown that at the present time the main tendencies of the progress of these processes are:

- Further development of the theory of the processes in the direction of complex modeling of physical and chemical processes occurring in metals as a result of its interaction with external sources.
- Development of technologies of surface saturation in multicomponent atmospheres.
- Usage of high energy sources for activation and intensification of thermo-chemical treatment.
- Development of combined technologies consisting of several classical methods of surface treatment for receiving of new combinations of properties.

New technologies of thermo-chemical treatment of steels are presented developed at the Department of Metal Science and Heat Treatment of MADI University (Moscow, RF) for the last 3 years. These technologies are based on nitriding process combined with other different methods of surface modification: laser alloying, thermo diffusion alloying, plasma treatment, galvanic and slurry metallization, oxidation, etc. There are several effective combinations of treatment; by regulation of parameters of each process it is possible to control the structure of surface layer on micro- and nano-scaled levels and to form coatings on various carbon and alloyed steels with required properties (hardness, wear and corrosion resistance, etc.) using in different conditions of operation of machine parts.

NEW PROCESS FOR ACCELERATED CARBIDE SPHEROIDISATION OF RST37-2 STEEL

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Conventional soft annealing of steel is normally carried out in continuous and batch furnaces with annealing times up to several tens of hours. The typical annealing temperature is just below AC1 but the soaking times are varied. The hold is followed by slow cooling to room temperature. During annealing, recovery of crystalline lattice takes place in ferrite matrix. Carbide spheroidisation occurs as well, i.e. the cementite lamellae transform into globular cementite. As a result, strength and hardness decrease, ductility increases and the ability of the steel workpiece to undergo plastic deformation is recovered. Normally, toughness and other dynamic response parameters grow significantly. The overall decrease in hardness in low-carbon and low-alloyed steels is governed primarily by the degree of softening of their ferrite matrix. The carbide spheroidisation alters, in most cases favourably, the dynamic response and plasticity properties of the material. In steels with higher carbon content (above 0.6%), carbide spheroidisation has substantial impact on hardness. The annealing process can be accelerated by temperature cycling around the transformation temperature AC1. However, according to literature sources, this process takes several hours as well. Annealing above AC1 and subsequent cooling is also possible. This can make the soft annealing process shorter but the time remains on the order of hours. The newly developed process consists in rapid temperature cycling close to AC1. This paper describes a process where this was achieved by induction heating. The experimental programme was conducted using plain structural steel RSt37-2. The carbide spheroidisation was explored and ferrite grain size measured by means of optical and electron microscopes. Mechanical properties, namely hardness, ultimate tensile strength, yield stress, elongation and impact toughness were tested. The heat treatment time has been reduced from several tens of hours required for conventional treatment to several tens of seconds. This offers potential time and energy savings.

SESSION D2 NON-FERROUS PROCESSES AND PHENOMENA

WEAR CHARACTERISTICS OF LIGHT WEIGHT SELF LUBRICATING AL-SI COMPOSITE

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In the present work investigations of wear properties of light weight self-lubricating Al-Si composites have been carried out. The composites are manufactured by the spray atomization and deposition technique. The reinforced particles used for the study is zircon sand, which is economical and easily available mineral. The self lubricating property of the material is catered by the addition of an immiscible element Sn, in the Al-Si alloy. The wear test has been performed on the pin on disc apparatus at constant velocity. Normal load and temperature were considered variables in the wear test. The wear rate of spray formed composite is significantly lower than the spray formed Al-Si alloy. The lubrication effect catered by the Sn addition further reduces the wear rate of composite. The microstructure of spray formed composite consists of fine size equiaxed grains.

The reinforced particles are equally distributed throughout the matrix of the composite. The as prepared samples were studied using SEM and X-ray diffraction techniques. The XRD results indicate the different phases present in the matrix. Hardness of the prepared samples has also been tested. Mode of wear mechanism has been analyzed from SEM micrograph of worn pin surface and collected debris during the dry sliding run. Composites are found to offer greater wear resistance than base alloy.

OXIDATION STUDY OF TI-AL-SI-C-N HARD COATINGS

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A hard Ti-Al-Si-C-N coating with Al contents ranging from 9% to 30% is deposited with the hybrid arc-enhanced magnetron sputtering (AEMS) technique. The oxidation study shows that the coating can hold good oxidation resistance up to 800 ^{o}C in air. As compared to Ti-Al-N coating which exhibits similar oxidation behavior and has been used in dry cutting tools, the wear resistance of Ti-Al-Si-C-N coating appears better at elevated temperature due to its relatively higher hardness (about 38 GPa) and lower friction coefficient (about 0.3) measured at room temperature, although both coatings show the identical tendency of hardness decreasing and grain size increasing with the temperature.

The XRD examination shows that the tested Ti-Al-Si-C-N coating holding of oxidation resistance till 800 ^{o}C is due probably to the formation of Al_2O_3 and TiO_2 particles in the coating surface, and therefore protects the coating from extensive oxygen penetration.

Keywords: Ti-Al-Si-C-N coatings; Microstructure; Oxidation resistance; Hardness

PLASMA TRANSFERRED ARC HARDFACINGS REINFORCED BY CHROMIUM CARBIDE-BASED CERMET PARTICLES

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Hardfacing is known as an effective method to enhance materials performance through combining properties of ductile metal matrix with high hardness of hardphases. In this work, multiphase metal matrix composite (MMC) coatings consistng chromium carbide and chromium carbide based cermets were produced using plasma transferred arc hardfacing (PTA) technique. Cr_3C_2 and Cr_3C_2-Ni powders mixed with Ni-based metal matrix and deposited on austenitic plates to minimise oxidation of substrate at elevated temperature.

The effect of Ni-binder on dissolution level of primary carbides during processing was studied. Furthermore, chemical pre-treatment of hardphases was applied to improve weldability of the precursor powders. Microstructure of materials was analysed with Scanning Electron Microscopy and Energy Dispersive Spectroscopy (SEM/EDS); phase distribution was characterised using X-ray Diffraction (XRD). Micro-mechanical properties, including hardness and elastic modulus of constituents, were measured by nano-indentation. The materials under consideration were characterized as tribo-materials by subjecting them to abrasive and impact conditions at temperatures up to 550℃.

Results indicated that Ni binder can significantly decrease dilution of the primary carbides during processing and improve the microstructure of the obtained coatings. Tribological tests revealed high wear resistance of the coatings at elevated temperatures, whereas chemically pre-treated Cr_3C_2-Ni based composites exhibited the lowest wear rates.

Keywords: tribology, surface treatment, high temperature wear, chromium carbide, hardfacing

TEMPERING AND HEAT TREATMENT EFFECTS ON METALLURGICAL PROPERTIES OF AN AL-BRONZE ALLOY, O.A EL-SHAHAT CENTRAL METALLARGICAL RESEARCH & DEVELOPMENT INSTITUTE (CMRDI) HELWAN , CAIRO , EGYPT.

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Aluminium bronzes are equally suitable for castings and wrought products such as rolled, forged, extruded and drawn products(1). Most of Cu-Al alloys were nor developed for heat treatment, nevertheless, their structures at the temperature of application are products of beta-phase transformation so that possibilities for heat treatment exist. Most attention has been given to the quenching and tempering procedures because this combination of heat treatment generally results in the highest mechanical strength(1.2) .

The cast aluminium bronzes are not commonly heat treated as the required properties can usually be obtained by careful selection of alloying element. Heat treatment is adopted on occasions for special purposes when exceptional combinations of properties are required(3).

EFFECT OF PHASE CONSTITUENTS OF ASTM F-1537 CO-CR ALLOY ON THE FORMATION OF S-PHASE DURING LOW-TEMPERATURE PLASMA CARBURISING

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Concerns over long term Co-Cr joint prostheses exist regarding wear of the articulating surface and the carcinogenic effect of the resultant metal ion release. It has been found in recent studies that surface-related properties as surface hardness and wear resistance of Co-Cr alloys can be improved by low temperature plasma carburising (LTPC) due to the formation of an S-phase layer on the surface. However, the characteristics of the surface S-phase layers produced by LTPC were found to be dependent on microstructure of the substrate materials.

This project was aimed at studying the effect of crystal structure (FCC and HCP) on the response of Co-Cr alloys to LTPC, thus advancing scientific understanding and establishing technology database for optimising the LTPC process. To this end, ASTM F-1537 Co-Cr specimens were high-temperature solution treated and aged to obtain different ratios FCC to HCP ratios. Low temperature plasma carburising was conducted using a DC plasma unit with a gas mixture of H_2 and CH_4 at temperature of 450°C. The phase constituents were quantitatively determined by XRD and EBSD methods. The microstructure of surface modified layers was characterised by optical microscopy, SEM coupled with EDX and EBSD and TEM. The hardness of the plasma carburised layers was measured on the surface and through cross-sections. Reciprocating wear tests in air were performed to evaluate tribological properties of the hardened layers. The results showed that the ratio of FCC/HCP phase in the Co-Cr alloy will greatly affect the microstructure, hardness, wear and corrosion resistance of LTPC treaded surface layers. This paper presents these new findings and the mechanisms involved are also discussed.

EVALUATION OF PACK ALUMINIZING OF DIFFERENT NI ALLOYS USED FOR THE PROTECTION OF COMPONENTS

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Parts of hot sections of gas turbines that operate at high temperatures can degrade due the oxidation. To improve the oxidation resistance of such components, which normally are nickel based alloys, coatings are used. These coatings can be obtained by diffusion techniques. Diffusion processes used for protection of components are strongly dependent on the chemical composition of the base material being coated. The primary target of pack aluminizing components is the formation of Nickel aluminides and it depends on the Ni and Al proportions (from the pack). Nevertheless differences in the aluminized surfaces and their behavior can be identified if used different substrates. The influence of chemical composition of two Ni alloys and processing parameters of pack aluminizing on the oxidation resistance are studied. Two temperatures (800ºC e 600ºC) and cementation time from 1h-16h are tested. Characterization of the aluminized surfaces include: optical, confocal and scanning electron microscopy, semi-quantitative analysis, Vickers microhardness and oxidation tests.

SESSION E1 MISCELLANEOUS PROCESSES

VACUUM HEAT TREATMENT OF SPRING STEEL 51CRV4

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The trend of the heat treatment of machine parts, including springs, is in the direction of vacuum heat treatment. Namely, cooling rates that can be achieved in modern vacuum furnaces with cooling in a stream of N_2, He or in a mixture of He and N_2 under the pressure up to 25 bar, already reach cooling speeds close to the cooling rates in oil. One can expect that the obtained microstructure and mechanical properties of springs after vacuum quenching and tempering will be comparable to those achieved by conventional heat treatment. In vacuum heat treatment, there is no risk of decarburization no formation of the oxide layer and no residues of quench oil on the surface. It offers than significant advantage when compared to conventional heat treatment. In the work possibilities of vacuum heat treatment of spring steel grade 51CrV4 are presented. Charpy-V notch (CVN) impact-test values are widely used in toughness specifications for spring steels, even though the fracturing energy is not directly related to the spring design. The plain-strain stress-intensity factor (K_{Ic}) at the onset of unstable crack growth can be related to the spring design; however, K_{Ic} values are not used in toughness specifications. This is surprising since to the designer K_{Ic} values are more useful

than CVN values because the design calculations for springs from high-strength steels should also take into account the strength and the toughness of materials prevent rapid and brittle fracture. An investigation was conducted to determine whether standardized fracture-toughness testing (ASTM E399-90), which is difficult to perform reliably for hard and low ductility materials, could be replaced with a non-standard testing method using circumferentially notched and fatigue-precracked tensile specimens. The results of this investigation have shown that using the proposed method it was possible to draw, for the normally used range of working hardness, combined tempering diagrams (Rockwell-C hardness - Fracture toughness K_{Ic} - Tempering temperature) for the vacuum heat treated spring steel grade 51CrV4. Fractografic and metallographic analysis of the K_{Ic}-test specimens used shows in steel the presence of positive segregations. It was found that the width of the segregations bands and the distance between segregations influence significantly the fracture toughness due to the presence of the bainite in matrix.

EFFECTS OF CRYOGENIC AND STRESS RELIEF TREATMENTS IN THE TEMPER CARBIDES PRECIPITATION IN AN AISI D2 TOOL STEEL

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The effects of cryogenic treatments and of stress relief treatment in the thermal cycle of a tool steel were studied. Cryogenic temperatures were: cryogenic (-196°C) and subzero (-80°C). The holding times at cryogenic temperatures were: 2, 3, 10, 24, 30 hours. The effect of including a stress relief heat treatment before the cryogenic treatment was also verified. Specimens submitted to different thermal cycles were studied using metallographic characterization (OM, SEM and SEM-FEG), X-ray diffraction and electric power measurements. No variation in the secondary carbides (micron sized) precipitation was found.

The temper secondary carbides (nano sized) were found to be more finely dispersed in the matrix of the samples with cryogenic treatment and without stress relief. The X-ray diffractions were carried out at the Brazilian Synchrotron Light Laboratory. It was verified that: i) lower austenite volumetric fraction was found in specimens submitted to subzero treatment if compared to specimens submitted to cryogenic treatments, an indication of a C curve kinetic behavior of the transformation; ii) the stress relief treatment produced a decrease in the c/a relationship of the martensite lattice parameters and an increase in the austenite a parameter (explained by carbon atoms partition from the supersatured martensite to the retained austenite); iii) after double tempering occurred an increase in the a parameter of the remaining austenite lattice, indicating an increase in the carbon content of the residual retained austenite, due to carbon partition; iv) the higher volumetric fraction of temper carbides (M7C3 and M2C) was found in specimens submitted to cryogenic treatment just after quench (to room temperature), followed by cryogenic treatment + stress relief, specimens just quenched and tempered and finally by those submitted to stress relief without cryogenic treatment. Thermo-electric power measurements (at INSA-Lyon) were used to follow the tempering at different temperatures and times. Cumulative isothermal treatments (130°C, 210°C, 350°C, 450°C e 520°C) with increasing times (1 minute to 130 hours) were used. It was verified that: i) the kinetic is favoured by the cryogenic treatment in the first temper stage (η or ε carbide precipitation); ii) the stress relief delayed the first and second temper stage; iii) the specimens not submitted to cryogenic treatment showed to have a bigger growing for the alloys carbides in the last stage of temper. The refinement of the temper secondary carbides was attributed to a possible in situ carbide precipitation during tempering: η -> θ -> M7C3 e M2C.

EFFECT OF DEEP CRYOGENIC TREATMENT ON PROPERTIES OF HS6-5-2 HIGH SPEED STEEL

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Deep cryogenic treatment (DCT) is a heat treating process consisting in long term holding of the steel part at temperature within the range of -175 to -196 °C. DCT is applied mainly for enhancing the service life of cutting tools. The aim of this work was to investigate the effect of deep cryogenic treatment on properties of HS6-5-2 high speed steel. Basic properties, such as hardness, resistance to wear, impact strength and bending strength, were measured and evaluated. Imaging of the microstructure of specimens was performed with SEM microscope. Research work allowed concluding that DCT followed by tempering leads to significant enhancement of toughness and wear resistance of high speed steel. Improvement of properties of steel is connected with continuation of transformation of over-cooled austenite, refinement of substructure of the martensite plates and presence of precipitations of fine carbides.

EFFECT OF DEEP CRYOGENIC TREATMENT ON DILATOMETRIC CURVE AND TRIBOLOGICAL PROPERTIES OF HIGH SPEED STEEL

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Advantages of deep-cryogenic treatment over standard heat treatment of high speed steels for the purpose of obtaining better exploitation properties are quoted in an increasing number of scientific papers to be found in literature. This paper deals with the improvements of high speed steel properties achieved by using deep-cryogenic treatment on test samples made of the PM S390 MC high speed steel. The effect of deep-cryogenic treatment on the dilatometric curve during tempering and on abrasive and erosion wear resistance has been investigated and compared with results obtained at a set of test samples of the same steel heat treated by conventional method (hardened and three times high temperature tempered). Dilatometric tests confirmed the existence of common processes in tempering, which depend on the initial state of steel (only quenched, and quenched and deep-cryogenically treated). These tests showed that deep cryogenic treatment had not completely eliminated the residual austenite, particularly in the case of the highest austenization temperature. The obtained tribological results confirm that the application of deep cryogenic treatment results in significantly improved erosive wear resistance, while the abrasive wear resistance depends on the applied temperature of austenization.

Key words: high speed steel grade PM S390 MC, deep cryogenic treatment, wear resistance

DEVELOPMENT OF ACTIVE LOW TEMPERATURE SALT BATH NITROCARBURIZING IN JAPAN

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Recent R&D trends and industrial activity in surface hardening of corrosion-resistant steels by nitriding and nitrocarburizing technologies are considered. Typical features of Japanese works show some differences compared with those of other countries. Most research deals with improvement of processing technologies and improvement of properties and of higher grade steels. Some companies use low temperature surface hardening technologies such as plasma carburizing, and gas nitriding and carburizing processes and supply their products to, for example, city water and components for various industrial applications. The low

temperature cyanate salt bath nitrocarburizing process is a promising technology and recent research has led to efficient low temperature nitriding.

In this paper, the general trends of R&D in the field and a new technology to measure the activity of SBN and their performance, will be presented.
Key words: Nitriding, Nitrocarburizing, Gas, Plasma, Salt bath, Stainless steels

SESSION E2 QUENCHING AND DISTORTION ENGINEERING

MEASUREMENT AND DATABASE CONSTRUCTION OF COOLING POWER OF QUENCHANTS BY USING SILVER PROBES

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The cooling characteristics during quenching of hot metallic parts depend on not only quenchant conditions and also the shape, size, material and surface condition of the parts. However, it is not easy to estimate the HTC on the surface of quenched complex-shaped steel parts like gears. Silver probes (rod, sphere, plate) have been used for evaluate the cooling characteristic in quenching, because of no surface oxidization and the high conductivity (sensitivity). In addition, heat transfer coefficient and heat flux which need for quenching simulation as thermal surface boundary condition can be calculated easily by using the lumped capacity method from cooling curve data of small silver probes. In order to establish a database of cooling power of different quenchants, cooling curve data of silver rod probes were measured by laboratory test method. Silver probes of 10 mm dia. x 30 mm were used for estimation of the cooling power of quenchants based on JIS K2242 and ASTM D7646-10 standards. Heat transfer coefficient data and heat flux data as a function of surface temperature were calculated from these cooling curve data by using the lumped heat capacity method or the inverse method. The DATABASE covers various quenchants for specified conditions. This report will show the detail and the availability of this database.

THE NUMERICAL INVESTIGATION OF THE EFFECT OF TRANSFORMATION PLASTICITY ON QUENCHING DISTORTION

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Transformation plasticity is one of the most important factors that cannot be neglected affecting the quenching distortion. The existing models describing transformation plasticity have been reviewed and the elastic-plastic constitutive relations for quenching process considering thermal strain, transformation strain and transformation strain has been discussed. The formulation in numerical simulation of quenching distortion has been introduced. The evolution of temperature field, transformation and stress/strain in the quenching parts has been analyzed based on two specific examples, and special attention has been paid on the final distortion with the introduction of different kinds of strain. The results show that the quenching parts of steels with different transformation characteristics may have totally different distortion modes even though the geometry is very simple, indicating the quenching distortion prediction a long term challenging task.

QUENCHING DISTORTION INDUCED BY INGOT SEGREGATION IN GEAR STEEL

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Quenching is widely used in gear steel to enhance the quality of mechanical products such as strength, surface hardness and service life. The quenching involves heating and cooling of wo rkpieces to obtain the desired physical and mechanical properties. Quenching distortion, main ly due to the plastic deformation induced by the thermal and phase transformation during que nching process, is inevitable. On the other hand, high precision is required for automobile gea rs in order to reduce gear noise and save the cost of grinding for eliminating the alterations in size and shape of automobile gears. Therefore, in recent year, in order to control and reduce t he quenching distortion of automobile gears, many works have been done to investigate influ ence of steel manufacturing process and heat treatment process on the quenching distortion of automobile gears. In this article, the influence of ingot segregation induced by the rectangul ar sectional shape of molds for continue casting on the quenching distortion of gear steel has been discussed.

In this study, in order to investigate the influence of the ingot segregation on the quenching di stortion, ring specimens with different diameters were designed. After ring specimens were tre ated by oil quench, the outer shape changes of various ring specimens were dertemined. It is indicated that the ring specimens made of the steel with square ingot segregation showes sy mmetrical distortion, and the ring specimens made of the steel with rectangular ingot segregat ion shows unsymmetrical distorion.

It was also found that the influence of the ingot segregation shape on the quenching distortion of ring specimens with bigger inner diameter, whose ingot segregation area was almost remo ved.

In a word, the research results show that the shape of the ingot segregation has strong influe nce on the quenching distortion for gear steel.

COMPARISON OF THE INTERFACIAL HEAT TRANSFER MECHANISMS AND REWETTING PROPERTIES OF VEGETABLE OIL AND PETROLEUM OIL- BASED QUENCHANTS AND POTENTIAL IMPACT ON QUENCH DISTORTION

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With the notable exception of an early reference published by Prof. Imao Tamura, the results of detailed examinations of the interfacial film formation comparison of vegetable oils and petroleum oils during the hardening of steel. This is important because the formation and breakage of any films formed at the hot-metal interface dictate whether heat transfer will occur by film boiling, nucleate boiling or convective cooling. This is particularly important because of the dramatic differences in the rate of heat transfer which affects the hardening ability of these oils as quenchants. Also important is knowledge of the rewetting properties of a quenchant not only from the perspective of hardening potential but also with respect to its ability to reduce distortion and the potential for cracking. This paper will discuss the results of a recently completed study to examine the relative difference of the heat transfer mechanisms of vegetable oils and petroleum oil-based quenchants using high-speed photography and videos of the quenching processes. The relative difference in the rewetting performance of vegetable oil and petroleum oil-based quenchants will also be discussed.

PREDICTION OF DISTORTION OF SIMPLE GEOMETRIES AS A FUNCTION OF FLOW FIELD AND ORIENTATION

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Racking of parts is a critical part of the heat treating and quenching operation. The application of the wrong method of racking can result in excessive distortion even when high quality quenchants are used. In this study, various simple geometries were examined at different orientations to quenchant flow using Computational Fluid Dynamics. The flow fields were determined, and the resultant heat transfer coefficients were mapped to the part surfaces. The expected part distortion was then determined using DANTE™. The resulting work serves as a basis for developing design rules for racking of parts to minimize distortion occurring from quenching

SESSION F1 THERMAL AND THERMOCHEMICAL PROCESSES

AGING AND MICROSTRUCTURAL DEVELOPMENT OF AL-LI(8090) ALLOY AND AL-LI COMPOSITES

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Different percentage of SiC particulates (0-15 wt.%) and Al_2O_3 particulates (0-10 wt.%) with Particle size of 40 µm were introduced into Al-Li (8090) alloy to produce composite alloys by using stir casting technique.

Mechanical deformation by rolling of 50% reduction in thickness was applied for some Al Li composites.

Microstructural studies of both Al-Li and Al-Li composites were carried out in cast and deformed conditions using optical microscope.

Also, aging kinetics of matrix alloy and Al-Li composites at 170ºC were investigated by using Vicker hardness measurements.

The results showed that the cast composites obtained some degree of porosity especially with high volume fraction of particulates. At the same time, the Al-Li composites showed some grain refinement compared with the matrix alloy.

The results of the aging studies showed higher peak hardness and lower peak aging times for both Al-Li 5, 10 wt % SiC and 5, 10wt% Al_2O_3 composites compared with the unreinforced matrix alloy. On the other hand, clustering of reinforcement particulates appeared to be responsible for reduced hardening of Al-Li 15%SiC composites.

Generally; aging result in considerable improvement of hardness for both matrix and composite alloys. This is attributed to the formation of both d' (Al_3Li) and S' (Al_2 Cu Mg) precipitates during aging.

TEMPERING CARBIDES AND MECHANICAL PROPERTIES IN HIGH ALLOY STEELS FOR HIGH TEMPERATURE APPLICATIONS

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High alloy steels are employed in various applications in industry, being some of them related to high temperature (>500°C) service conditions. Hot work tools and special materials for power plants are the most important example in this field. At such conditions, strengthening mechanisms are dependent on the solid solution and mainly on precipitation hardening.

Recent changes in both chemical composition and processing conditions have promoted the development of new steels for such application, which distinct combinations of strength and toughness. Reports in literature have shown that Si, although not a carbide forming element, influences secondary tempering carbides distribution, based on Cr and Mo alloying elements. In the present paper, different compositions of tool steels are compared, in terms of tempering precipitates and their interrelationship to the mechanical properties. Due to the nanoscale size, carbides were evaluated by transmission electron microscopy, in thin foils and extraction replicas. Mechanical properties were based mainly on hardness and impact toughness data.

All evaluations were performed in the hardened and tempered condition. The results point that large carbides of M7C3 type are present in the steel microstructure after high temperature tempering, being their distribution related to the final toughness. In terms of high Mo steels, undissolved carbides (during austenitizing) of M3C type are observed in the final microstructure, due to the stabilization with Mo. On the other hand, the dissolved part of Mo is related to the formation of small M2C needles, important for precipitation hardening. Therefore, the amount of Mo trapped in M3C after quenching is pointed as a new possible source for better strength from precipitation hardening.

HEAT TREATMENT OF HIGH CHROMIUM CAST IRONS - THE IMPORTANCE OF MICROSTRUCTURAL CHARACTERIZATION.

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High Chromium Cast Irons are used for their abrasion resistance in applications such as liner plates and grinding media in tube mills, crushing rollers and tables in vertical spindle mills and the impellors and bodies of slurry pumps. Before service the cast wear parts are normally subjected to heat treatments including softening for tool machining, destabilization, air hardening and tempering. The physical metallurgy underlying the development of cast microstructures in these irons, and their subsequent structural modification by heat treatment is relatively complex. Hence structural characterization via electron microscopy has an important role to play in furthering our understanding of the phase transformations that control the microstructures and hence the service behaviour of these irons. This paper shows how both Scanning and especially Transmission Electron Microscopy and associated micro-analytical techniques can provide valuable information on cast microstructures and, in particular, about the effects of thermal treatments on the nature of eutectic and secondary carbides and on the matrix structures in these irons.

INFLUENCE OF SALT BATH TEMPERATURE, AGITATION LEVEL AND WATER ADDITION ON ITS COOLING RATE

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Salt bath technology, at this time and age, is still vital and irreplaceable one. In past, oftenly compared to baths of molten metal with low melting point (like led), superiority of salt baths is

well justified and not in question today. In comparison to molten metal, molten salts have up to ten times higher specific heat capacity, and much lower viscosity.

Although quenching power of molten salts is (as given) poor, certain factors can be taken into account to have the cooling rate modified within relatively wide limits;

- According to Newton's law, the temperature of the salt bath may be decreased in order to have cooling rate increased;
- Since heat transfer occurs through convection, changes in the rate of agitation will influence cooling rate of the salt bath;
- And, the most effective factor of cooling rate changes (increase) is an addition of water.

This paper is intended to give a detailed model of how individual factors and their combinations influence cooling rate of a salt bath. A special effort has been invested in explaining the interaction of the agitation rate and the addition of water; hence the addition of water increases quenching power directly, as well as indirectly by reducing molten salts viscosity and allowing it to flow more freely. Precise regulation of any parameter given, being done by use of modern technologies, made data retrieved ever so accurate. Experimental work was done at the premises of the Laboratory for Heat Treatment, Faculty of Mechanical Engineering and Naval Architecture, by utilizing proprietary molten saltpeter salts (potassium/sodium nitrate) and a probe acquired for that sole purpose. The data obtained from the probe (and its belonging software) was then processed by Design Expert software solution.

HARDNESS PROFILE IMPROVEMENT OF PLASMA NITRIDED HIGH-SPEED STEEL BY GLOW DISCHARGE HEATING

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The hardness of a plasma nitrided high-speed steel surface is very high at the very surface by the formation of nitrides and diffusion layer and degrease steeply to the value of substrate. Such brittle surface and steep change of the hardness are not favorable for tool applications because of the tool chipping. In this experiment, the improvement of such hardness profile of plasma nitrided high-speed steel by glow discharge heating as the post nitrided diffusion treatment was investigated.

High-speed steel was plasma nitrided at 500 degree C under 667 Pa in gas mixture of nitrogen and hydrogen for two hours. Then a diffusion process in the same chamber was carried out by change the gas to hydrogen or argon. The plasma nitrided high-speed steel also diffusion treated by furnace heating. The microstructure and the hardness profiles of these two post-nitrided treated samples were compared.

The diffusion treatment by glow discharge heating makes the hardness gradient from steep to gentle more effectively than by furnace heating. Furthermore, the hard nitride layer was eliminated by glow discharging, but the layer remained after furnace heating. It was found that the effect of post nitrided diffusion treatment by glow discharge heating is due to the sputtering effect and the acceleration of diffusion by ion impaction.

NITRIDING EFFECT ON FATIGUE RESISTANCE OF GRAY CAST IRON

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This work aims to study the influence of nitriding on fatigue resistance of circles bars made with gray cast iron. For this study was taken three chemical compositions. The selected materials include a gray cast iron with mixed initial microstructure (martensite plus bainite), a martensitic gray cast iron and a martensitic gray cast iron alloyed with niobium. These materials were gas nitrided for 3 and 6 hours. The relationship between the mechanical behavior and the treatment time (depth of nitrided layer) was established. Samples were tested under a cyclical bending load. The stresses was obtained in the cross section, where was performed the biggest moment. Results were statistically treated by means of Weibull analysis. The Whölers diagram was obtained for each studied material. Nitriding showed that fatigue resistance on gray cast iron with martensitic structure is higher when compared to that with mixed structure. The depth of nitrided layer has not influenced the fatigue limit.

SESSION F2 PROCESSES AND PHENOMENA

WEAR RESISTANT THERMAL SPRAYED COMPOSITE COATINGS BASED ON IRON SELF-FLUXING ALLOY AND RECYCLED CERMET POWDERS

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Thermal spray and WC-Co based coatings are widely used in areas subjected to abrasive wear. Commercial hardmetal thermal spray powders for HVOF are relatively expensive.

Therefore applying these powders in cost-sensitive areas like mining and agriculture are hindered. Nowadays, the use of cheap iron based self-fluxing alloy powders for thermal spray is limited. The aim of this research was to study properties of composite powders based on self-fluxing alloys and recycled cermets and to examine the wear resistance of thermally sprayed (HVOF) coatings from composite powders based on iron self-fluxing alloy and recycled cermet powders (Cr_3C_2-Ni and WC-Co). To estimate the properties of recycled cermet powders, the sieving analysis, laser granulometry and morphology study were conducted. For deposition of coatings High Velocity Oxy-Fuel spray was used. The structure and composition of powders and coatings were estimated by SEM and XRD methods. Abrasive wear (abrasion, erosion and impact wear) performance of coatings was determined and compared with wear resistance of coatings from commercial powders.

The wear resistance of thermal spray coatings from self-fluxing alloy and recycled cermet powders at abrasion is comparable with wear resistance of coatings from commercial expensive spray powders and may be an alternative in tribological applications in cost-sensitive areas.
Keywords: recycled, composite coatings, HVOF spray, hardmetals, wear resistance

ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY (EIS) FOR THE DETERMINATION OF STEEL PARTS SURFACE CONDITION

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Surface reactions during a thermochemical treatment require clean, and non-passive surfaces, enabling diffusion of reactants into the bulk material, e.g. in the process of gas nitriding. Clean surface condition is achieved by cleaning, and surface activity by activation. The success of the cleaning process depends on the condition of the cleaning baths and rinsing agents. After the heat treatment, a wear-resistant hard surface layer is obtained. The layer is supposed to be of uniform thickness, without any defects or "soft spots". In addition, corrosion resistance is a demanded property. Thus there are three areas where information on the condition of either surfaces or media is needed for improved process control, and where Electrochemical Impedance Spectroscopy (EIS) is in principle able to deliver that information. The measuring technique enables to determine Ohmic and non-Ohmic resistances in an electrochemical cell, where the surface to be analyzed forms the "working electrode". The resistances are measured as frequency-dependant impedance in an AC circuit. Working and counter electrode are in electrical contact via an electrolyte. If surface condition is to be detected, the electrolyte is a non-corrosive buffer solution. The impedance of the circuit represents the electrical properties of films and layers on the working electrode's surface, i.e. contamination films and/or passive reaction layers.

Similarly, the EIS method can be applied to determine the integrity and the corrosion resistance of a compound layer after, e.g., nitriding. Here, electrolytes with different degrees of corrosivity can be applied to study the hard layer's chemical stability.
If the measuring probe is replaced by a conventional conductivity cell, the electrochemical properties of an electrolyte, i.e. a cleaning or rinsing agent, can be recorded. These properties are composition- and concentration-dependant, so that the condition of these media can be monitored.

Although EIS looks like a universal measuring technique is not yet used in industry. One reason is the lack of proper measuring probes (in the case of surface measurements), another the need for interpretation of the complex measuring results.

INFLUENCE OF DYNAMIC DEFORMATION ON MICROSTRUCTURE TRANSFORMATIONS IN AUSTEMPERED DUCTILE IRON

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The article analyzes changes in ADI (Austempered Ductile Iron) microstructure under the effect of dynamic surface deformation. Investigations covered the cast iron characterised by a tensile strength of 1049MPa and elongation of 16% with high austenite content in microstructure. The task to be performed consisted in FEM simulation of the deformation effect, using a ø10mm ball pressed dynamically into the examined material with experimental verification of the obtained results. The ADI microstructure was examined by LM, SEM, and XRD technique. The kinetics of the deformation-induced transformations was also analyzed.

The presence of metastable austenite was observed along with the occurrence of a deformation-induced martensitic transformation. The developed model may serve in evaluation of the shotpeening effect on the condition of microstructure in surface layers of the austempered ductile iron.

STUDY OF DUCTILE FRACTURE BEHAVIOR OF AL-BEARING DUPLEX STAINLESS STEELS

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The properties of ductile fracture are generally evaluated by elongation, reduction of area in tensile test and absorbed energy in Charpy impact test. However, it is estimated that the effect of metallurgical factors such as precipitates and microstructures on these properties of ductile fracture depends on varius method of tests. In this study, the tensile test and the instrumented Charpy impact test were carried out in JIS SUS329J3L, where the amount of Al is varied from 0.014 to 0.084 mass%. The round bar specimens were used for tensile test and the instrumented Charpy impact test was standardized by JIS Z2242. In all steels, the volume fraction of ferrite phase were 70%. Amount of Al precipitates(AlN and Al_2O_3) and its diameter decreased with decreasing Al content. To examine the mechanism of ductile fracture in round bar tensile tests, the reduction of area was measured by a three dimention shape constructive method, and the relationship between the reduction of area and distance from the fracture surface calculated by using it. The reduction of area curve is slightly changed by changing the Al content.

The local deformation energy was calculated by load-displacement curves which were obtained from the tensile tests. The local deformation energy changed with decreasing in Al content. However, crack propagation energy of instrumented Charpy impact test increased largely with decreasing Al content compared with tensile test. The specimen which used the tensile test and the instrumented Charpy impact test were fractured by crack propagation which was produced by the void nucleation, growth and connection. Therefore, the difference of these energies means the difference of the void nucleation, growth and connection processes. In the tensile tests, void nucleation usually occurs at the beginning of local deformation, on the other hand, in the instrumented Charpy impact tests, void nucleation occurs with crack propagation. The fracture surface in tensile tests exhibits type IIvoids while that in Charpy impact test exhibits type I voids.

These difference in both tests indicates characteristics of ductile fracture. In addition, growth and connection depends on condition of precipitates as well as the void nucleation. It is elucidated that difference of ductile fracture is due to the change in Al content.

DUCTILE FRACTURE MORPHOLOGY AND VOID FORMATION IN LOCAL DEFORMATION FOR FERRITIC STEELS

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In ductile fracture, the local deformation is related to the nucleation and the growth of the voids and the formation of the fracture surface. In this study, we examined that relationship between the local elongation in tensile test and the amount of precipitates for chromium bearing ferritic steels. By coarsening of precipitates in the steels used, the distance between the precipitates spread, because quantity of precipitates was the same levels in these steels. As the interparticle spacing spread out, the 0.2% proof strength was decreased and the local deformation energy. On the other hand, the effect of the precipitates diameter on the uniform elongation can not be clearly observed.

By observation of a cross section for each specimen after tensile test with AsB detector which is the good way to observe voids on SEM, it was revealed voids exists widely from a fracture surface so that the local elongation was increased.
The precipitates cause the morphology change of ductile fractured surfaces. The fractography of the fracture surfaces unveiled that the coarsening of precipitates caused an increase in the diameter and a reducing in the depth of a dimple unit. After the 5-line profile of a fracture surface, the line profile is used for evaluating quantitatively the surface texture by Fourier function. The fracture surface texture was divided into many components by the function. This Fourier analysis revealed that the precipitates influenced on the texture of fractured surface. The relationship between the amplitude of wavelength of the fracture surface and the

interparticle spacing was found to be linear. This finding that the growth and connection process of the voids varied according to the interparticle spacing.

The main results obtained in this study are summarized as follows;

1) The local elongation was increased by the precipitates diameter.
2) The difference of the interparticle spacing changes the distribution of the void.
3) The Fourier analysis of the fracture surface revealed that the local deformation energy is influenced by the fracture surface morphology the rough the voids formation process.

SESSION G1 QUENCHING AND DISTORTION ENGINEERING

ULTRASONIC ASSISTED WATER QUENCHING OF ALUMINIUM AND STEEL CYLINDERS

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Because of high mean quenching rates and easy realisation, quenching of metallic components during heat treatment is often done in liquid baths (e.g. water, oil). If the boiling temperature of the liquid is lower than the initial temperature of the component, liquid quenching will proceed in the three stages of film boiling, nucleate boiling and convection. These stages exist locally and temporally non-uniform on the component surface and cause very different heat transfers. In particular the heat isolating vapour film causes an undesirable non-uniformity of the quenching step. As a result, distortion and residual stresses may occur. The application of ultrasonic fields is one possibility to affect liquid quenching.

This method allows the destabilization of the vapour film and causes an accelerated and more uniform quenching process. The matter of this study is the cooling power characterisation of ultrasonic assisted compared to conventional water quenching of steel (X5CrNi18-10) and aluminium cylinders (AlSi1MgMn). For this purpose, an apparatus for a defined ultrasonic assisted water quenching was developed, which offers the possibility of varying numerous process parameters. The ultrasonic amplitude, the size of the sound emitting surface (sonotrode) and the distance between sonotrode and sample as well as the water temperature were varied during ultrasonic assisted quenching. The cylinders were equipped with several thermocouples near the surface in circumferential and axial direction. In dependence of the different process parameters the ultrasound causes a significant destabilisation of the vapour film and hence an increase in cooling rate in front of the sonotrode. The cooling rate in front of the sonotrode increases with an increasing size of the sound emitting surface, with a decreasing distance between sonotrode and sample and with an increasing ultrasonic amplitude. Using one sonotrode, a more homogeneous cooling has been observed in axial direction, but - as expected - an inhomogeneous cooling in circumferential direction. It is intended to enhance the homogeneity in circumferential direction by using more (at least two) sonotrodes.

CHARACTERIZATION OF WATER BASE COPPER NANOQUENCHANTS BY STANDARD COOLING CURVE ANALYSIS

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Water base copper nanofluids having concentrations varying from 0.001-0.1vol% were prepared and used as quench media for immersion quenching. Cooling curve analyses were

carried out by using standard ISO/DIS 9950 quench probe. An inverse heat conduction model is employed to estimate the metal/nanoquenchant interfacial heat flux transients from the measured temperature field and thermo-physical properties of the quench probe material. The addition of nanoparticles had a significant effect on the cooling performance of the quenching media. Quenching in nanofluid shows longer vapour blanket stage as compared to water. Further, all the six cooling curve parameters were found to be altered by addition of nanoparticles to water. The heat flux curve shows a maximum shortly and then drops rapidly during quenching. The peak cooling rate and heat flux of water are increased by addition of very low volume fraction of nanoparticles. Both parameters decreased with increase in concentration of nanoparticles. The results suggest that the severity of cooling of the water could be altered by addition of copper nanoparticles.
Keywords: cooling curve analysis, copper nanoquenchants, cooling rate, heat flux transients

THE EFFECT OF TIO2 NANOPARTICLES ON FLUID QUENCHING CHARACTERISTICS

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Nanofluids are colloidal suspensions of nanoparticles in base fluids. Some of the particles used in recent research are metal oxide and carbide particles, such as SiC, CuO, Al2O3 and TiO2, graphite and carbon nanotubes and particles. Quenching in such colloids results in better cooling abilities, higher impact toughness and smaller dimension changes of steels, compared with pure quenching media.

In this research nanofluids with TiO2 powders of 50 nm average particle size were investigated. Base fluids of primary interest were deionized water, some commercial quenching oils and Polyalkylene Glycol (PAG) water solution of various polymer concentrations, 5 - 30 %vol. The investigated fluids were prepared by the addition of the same TiO2 powder with different concentrations - from extremely low, 10 mg of nanopowder per litre to 1 g/l. The cooling characteristics of these colloids were compared with the results of base fluids but also with the previous measurement results carried out in the fluids with addition of Al2O3 micron and submicron size particles.

All of the cooling curves were measured and recorded by IVF smart quench system using stainless steel probe with 12.5 mm diameter in accordance with ISO 9950 standard. Cooling rate vs. temperature and time was compared for all of the investigated media. Preparation of fluids was conducted with great care, with respect to mechanical stirring and sonification times. Average particle size was measured for each case. To better understand the phenomena that occur during quenching process high speed camera was used for recording of the experiments.

Titanium oxide nanoparticles show most significant effect on the cooling properties of water based polymer solution. With higher particle concentration maximum cooling rate increases. When it comes to quenching with nanoparticles suspended in oil, cooling rate at full film stage is higher thus shortening the stage period. The results show that maximum cooling rate is almost the same for various particle concentrations. When comparing the results of experiments with alumina particles, smaller and thermally less conductive TiO2 particles show greater effect on nanofluid properties.

Keywords: nanofluid, quenching, TiO2 nanoparticles

PROPERTIES OF DIFFERENT COMPOSITION OF OILS AS QUENCHANTS

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Quenching is the most common method of hardening metals, which involves continuos cooling from given austenite temperature to achieve fully transformation into martenzite structure. The cooling rate mainly depends on thermal characteristics of the metals, section thickness of the workpieces and the quenching medium heat-removal properties. The oils as quenching media contain base oil and different types of additives according to application requirements. As the base oil, it is possible to use mineral oils, synthetic and natural oils separately or in combination. Mineral base oils are widely used because of their advantages in stability in comparison to natural oils, or lower prices in comparison to synthetic oils. There are numerous compounds available for use but, besides of functional properties, the environmental and safety requirements have a strong influence on the components selection. Based on that the barium additives must be replaced with less harmful compounds. Petroleum derivatives should be changed with renewable base stocks that are biodegradable, as general trend in lubricant development. By changing the composition of quenching oils, base oils and additives, heat transfer characteristics are also changed. In this study are presented the examination results of physical-chemical properties and also cooling characteristics of new quenching oils with different compositions. Cooling curves for different compositions have been evaluated according to ISO 9950 standard.

SESSION G2 EXISTING AND EMERGING SURFACE ENGINEERING PROCESSES

LOW-TEMPERATURE ACTIVE-SCREEN PLASMA SURFACE ALLOYING OF MARTENSITIC AND PRECIPITATION HARDENING STAINLESS STEELS

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Both martensitic stainless steel (MSS) and precipitation hardening stainless steel (PHSS) are widely used in many industrial sectors because of their good corrosion resistance, superior mechanical properties and adequate wear resistance for some applications. However, their wear resistance needs to be improved for some demanding tribological applications. Active screen plasma surface alloying using low treatment temperatures can cause the formation of a new phase in the stainless steel, commonly known as the S-phase. This new phase has proven to have superior tribological properties along with very good corrosion resistance.

The major aim of this work is to investigate the feasibility of forming S-phase layers in martensitic (440C) and PH (17-4 and 17-7) stainless steels by low-temperature active screen plasma surface alloying with nitrogen (nitriding) and carbon (carburising) to improve their corrosion-wear properties. To optimise the most suitable active screen plasma process parameters for MSS and PHSS, specimens were treated at different temperatures starting from 350 ℃ to 450 ℃ for 20 h time duration in an atmosphere of 25% nitrogen balance hydrogen (nitriding) and 1.5% methane balance hydrogen (carburising) under 0.75 mbar pressure using 5% substrate bias. A phase analysis of the nitrided and carburised layers was carried out using X-ray diffraction technique.

Chemical composition especially nitrogen and carbon depth profile measurements were obtained using glow discharge optical emission spectroscopy (GD-OES), whereas microhardness and load bearing capacity tests were performed using Vickers hardness tester. Scanning electron microscopy (SEM) technique was employed to observe the diffusion layers as well as the indented cracks generated during load bearing capacity tests. Corrosion resistance of the plasma surface alloyed stainless steels was evaluated by salt spraying, immersion and electrochemical testing whereas wear resistance was measured using TE79 tribometer under reciprocating sliding conditions. It has been found that plasma treatment

temperature can significantly affect the mechanical as well as tribological properties of MSS and PHSS.

MODIFICATION OF HOT WORK TOOL STEEL SURFACE BY DIFFERENT THERMOCHEMICAL TREATMENT

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The surface of the hot work tool steel W300 grade was subjected to different thermochemical treatment in order to improve mechanical properties, corrosion and wear resistance. The nitrocarburising with or without post oxidation based on TENIFER procedure and low temperature powder aluminising with or without post oxidation was performed.

Nitrocarburised layers were characterized by testing their basic properties (compound layer thickness, nitrocarburising depth, surface hardness) as well as their corrosion resistance. The microstructures of aluminised samples were analysed by means of a Scanning Electron Microscope (SEM). Glow Discharge Optical Spectroscopy (GDOS) and Energy-Dispersive X-ray spectroscopy (EDX) were employed to investigate distribution of elements in the coating layer. Lastly, a corrosion resistance of aluminised samples were examined.

LOW AND HIGH TEMPERATURE PLASMA NITRIDING OF AISI F51 DUPLEX STAINLESS STEEL

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In this work an AISI F51 duplex stainless steel was DC-Plasma nitrided (PN) at 400^{o}C and 550ºC, during 20 hours in a 75% N_2 + 25% H_2 atmosphere. The 400ºC plasma nitrided specimen showed a modulated nitrogen rich expanded austenite (γ_N) layer formed on the specimen´s surface. The expanded austenite layer observed on top of the ferritic regions was 3 µm thick, while an expanded austenite layer 2 µm in thick was observed on top of the austenitic regions. Very fine martensite needles were observed in the layer formed on top of the ferritic regions.

For the 550ºC PN treatment the nitrided layer was 13 µm thick in the austenitic regions and 18 µm in the ferritic regions. CrN, Cr_2N and Fe_4N nitrides were identified by X-ray diffraction. A carbon enriched layer 18 µm thick was also observed beneath the nitrogen rich nitrided layer.

A strong hardening effect with surface hardness reaching up to 14 GPa was observed in the specimens PN at 400 and 550ºC. For nitriding at 400^{o}C the surface hardening can be related to the formation of a highly stressed nitrogen expanded austenite layer, with lattice parameter close to 3.72 nm, indicating a nitrogen concentration ~12 at% N. For nitriding at 550^{o}C hardening is related to the fine and homogeneous nitrided precipitation observed in the diffusion zone.

EFFECT OF SURFACE CLEANING ON HEAT TREATMENT RESULTS

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A cleaning process is not only required ***subsequent*** to quenching of heat-treated parts for the removal of quenching medium residues from the components' surfaces, but also ***prior*** to heat treatment, for the removal of residues from metal working, anti-corrosives or soil from storage an transport. Surface condition and cleanliness affect the heat treatment result, especially in thermochemical processes which require active surfaces, free from passive films or diffusion barriers.

Lubricants used in metalworking, water-based cleaning agents and their chemical components have been researched for their ability to hinder or prevent the formation of hard layers in a thermochemical diffusion process like gas nitriding. Specimens were gas nitrided after applying a layer of contaminants. Analytical tools were hardness measurement, microscopy, and surface analysis, combined to reveal kinds of interaction between manufacturing residues and heat treatment processes.

Based on general demands on workpieces' cleanliness and on specifications by heat treatment, cleaning processes, agents and components of cleaning plants are discussed. General demands refer to the residue-free removal of contaminants, even from complex geometries, comprising cleaning agent residues and water ingredients with respect to the type of water (e.g., water hardness). In addition, the cleaning agent itself must not leave any residues on the surfaces after the cleaning process is finished.

INVESTIGATION OF THE MASS TRANSPORT UPON LOW PRESSURE CARBONITRIDING

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The development and the realization of concepts to optimize engines in terms of performance and consumption poses a higher thermo-mechanical load on the parts, e.g. on diesel injection components through further pressure increase. These steadily increasing requirements can be realized with high-strength, but cost-intensive steels. An encouraging alternative to achieve higher temperature resistance is the application of the new heat treatment process low-pressure carbonitriding of low alloyed steels.

However, a solely experimental process development of low-pressure carbonitriding for a given component geometry is very cost extensive. A simulation based process development would be preferable. Simulation requires a process model which needs to be based on a fundamental understanding of the process background including especially modeling the mass transport to predict the carbon and nitrogen concentration profiles.

Within the project CARBONIT (support code: 03X3522A), which is funded by the German Federal Ministry of Education and Research, low-pressure carbonitriding for mass production is studied and developed under the consortial leadership of the Robert Bosch GmbH. The goal of the Robert Bosch subproject is to predict the integral carbon and nitrogen uptake and the respective concentration profiles in complex geometries. This will result in an adequate mathematical characterization of the low-pressure carbonitriding process, wherein complex transport mechanisms as well as gas and surface reactions run simultaneously.

To characterize the mass transport the process is divided into the following steps:

- thermal decomposition of carbon and nitrogen donors,
- gas mass transfer to the steel surface,
- adsorption of the carbon and the nitrogen donors,
- desorption of nitrogen
- chemical reactions on the steel surface,
- diffusion of carbon and nitrogen inside the steel,

- effusion of recombined nitrogen.

The influencing process parameters for solubility, diffusion and effusion were defined and experimentally investigated. Firstly the influence of the alloying composition on the solubility of the gases was investigated. In the next step the diffusion processes were modelled applying the calculation method for the diffusion coefficients proposed Slycke. With an additional pre-factor for the diffusion coefficient a good correlation between experimental and simulated nitrogen concentration profiles was achieved.

The modelling approach has been validated by experiments in a laboratory scale setup and in an industrial vacuum furnace. The rate coefficients for decomposition reactions, for desorption, adsorption, solution, effusion and diffusion were determined by a parameter study. The mathematical model allows a good prediction of concentration profiles within the framework of accuracy of measurements.

INVESTIGATION OF PHASE COMPOSITION OF HIGH-TEMPERATURE CHROMIUM STEELS AND THE CHROMONITRIDING PROCESS IN AUSTENITIC ALLOYS

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Creep resistance of 9% chromium high-temperature steels is determined by the alloy content and structure resulting from heat treatment. This paper describes analysis of the phase composition using CALPHAD numerical modelling methods of known modifications of 9% high-temperature chromium steels.

On the basis of calculated data and experimental results a composition for new high-temperature steels with additional Co alloying (up to 3%) and varying carbon contents in the range 0.02-0.10% is proposed. Results are shown for investigations on high-temperature chromium steels.

On the basis of complex laboratory investigation and industrial pilot heats, optimal composition variants for alloy content of high-temperature Cr-Mo-V-Co steels for practical application have been determined.

CNIITMASH has developed a chromonitriding technology for improving the corrosion, wear and scratch behaviour, as well as protection from self-welding and other service characteristics of austenitic steel components and nickel alloys. The technology is intended for strengthening of valves and bushings, water pump components, and components operating in liquid metal, burnt fuel residue, and other aggressive environments.

Thermodynamic analysis of phase formation conditions during the chromising and subsequent nitriding process over a wide range of temperatures and saturating media has been carried out. The technology has been optimised for process and media composition leading to a structure with maximum surface properties.

SESSION H1 MULTI-SCALE MODELLING OF SURFACE SYSTEMS

MOLECULAR DYNAMICS SIMULATION OF THE ADSORPTION OF ATOMIC TI AND N ON TIN SURFACE (SESSION H1)

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B1 structure TiN exhibits excellent mechanical and distinctive physical properties. It has been widely used as variouse protective coatings and functional coatings. The TiN film growth during variouse vapor deposition technologies is primarily attributed to the fluxes of atomic N and Ti, which react on the surface of substrate. Therefore, the adsorption of incident atoms on the surface of solids is an important step in the film growth, but little work was done about it due to the difficulty of experiments. A molecular dynamics model was built to describe the collision of incident Ti and N atoms with TiN surface. With this model and a recently developed potential for the Ti-N system based on the second nearest-neighbor modified embedded-atom method, the dependences of the adsorption of atomic Ti and N on TiN surface on the kinetic energy of atoms, the direction of incidence, the temperature of the substrate and the degree of coverage were studied. The results show that the sticking coefficient of atomic N has higher dependence on deposition condition than that of atomic Ti, and the incident energy plays the most important role on the adsorption.

VIRTUAL GRADED MICROSTRUCTURE GENERATION FOR CRYSTAL PLASTICITY ANALYSIS

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A novel grain structure modelling scheme is presented to generate virtual graded grain structures for crystal plasticity finite element (CPFE) analyses. A virtual graded grain structure consists of a group of grain aggregates with distinct or gradually changing grain morphological properties, including both the mean grain size and the grain size distribution. Virtual graded grain structures are useful in simulating, for example, thin films with the crystal structures of both film and substrate explicitly modeled and the interface morphology matching that of an actual coating, i.e. film/substrate interfaces which are locally non-planar due to the grain structures, roughness of the substrate surface and/or interdiffusion between film and substrate during deposition. Other structures such as heat-affected zones in welds can also be simulated using this technique. In practice, a graded grain structure can be divided into a set of sub-regions, denoted as multiple zones, with approximate homogeneity in each sub-region. The proposed scheme employs the controlled Poisson Voronoi tessellation (CPVT) model, which is capable of generating grain structures with regularity and grain size distribution control, to generate the grain structure for the individual zones. An additional module has been developed to generate grain seeds within the interfacing area between two adjacent zones. Based on this scheme, a grain structure can be constructed such that the virtual grain structure is statistically equivalent to the expected grain size distributions, which are specified by the mean grain size, a small grain size, a large grain size and the percentage of grains within that range. A software system (VGRAIN) has been developed implementing the proposed multi-zone CPVT model to generate graded grain structures for crystal plasticity finite element (CPFE) analysis. To demonstrate the proposed scheme and the VGRAIN system, plane strain CPFE analyses of the indentation of graded thin film microstructures are presented.

GENERIC MODEL FOR THE ANALYSIS OF THE FAILURES OF MULTILAYERED COATINGS ON TOOLS AND ENGINEERING COMPONENTS (SESSION H1, M3-2S)

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Efficiency and effectiveness of design and analysis of the hard coatings and corresponding manufacturing processes are not satisfactory although they have been deployed in industries for many years. This is especially the case when multi-layered nano-coatings on tools or engineering components are to be analysed. One of the key issues to be addressed concerns many failure-mechanisms needing to be considered for a complex multi-layered coating and surface system. Typical failure-mechanisms responsible for the tool damage, for example,

include fracture, delaminating, wear and thermal and mechanical fatigue. A generic model which is able to take most of the key factors associated with failure-mechanisms into account is what needed from industrial design and analysis point of view. In the M3-2S project an innovative modelling strategy and analysis procedures have been developed in order to produce a generic model to satisfy this requirement. As a result, the model can act as an efficient tool for the design and analysis of coated-surface systems and corresponding manufacturing processes. Key features of the model and associated modelling procedures include:

- (i) FE-based parameterised modelling allows for easy modifications of design and process parameters for analysis iterations, and hence, for easy facilitation of sensitivity analysis and design optimisation;
- (ii) Residual-stress prediction using a newly-developed theoretical model, considering the atomistic-scaled lattice mismatch as a main contributor to the residual stress during the deposition process;
- (iii) Thermo-mechanical coupled modelling allows for the consideration of the thermal effects during coating and under the service conditions;
- (iv) A cohesive-zone model allows for the prediction of crack initiation and propagation within the coating-layer subjected to the various types of loadings, where the critical energy release rate is calculated using the atomic First Principle, allowing for the effect of the atomic structure on the material properties being taken into account during the analysis;
- (v) A sub-modelling procedures enable the scaling down of the model-geometry from the macro-scale to the micro/nano scale to enable detail analysis and a link to the micro-mechanics modelling;
- (vi) A cohesive law under cyclic loading enable the analysis of the fatigue damage behaviour of the coating surface under the cyclic loading;
- (vii) A wear model is being developed for the prediction of the wear rate under the cyclic loading.

The proposed paper intends to report fundamentals and numerical procedures associated with the development of the generic model described above, followed by presenting experimental-validation cases and conclusions.

CUTTING SIMULATION – BASIS FOR COATING-SUBSTRATE-OPTIMIZATION (M3-2S)

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Compared to standard machining procedures, workpieces in high-performance cut-ting are machined at higher speeds, with larger loads and at a higher feed rate. The aim is to achieve a large chipping rate. Compared to straightforward high-speed cut-ting (HSC) – that is, machining at a high cutting rate – HPC generally involves lower cutting speeds and significantly larger cut depths. In addition to the machining pro-cess itself, HPC generally also integrates all the other factors involved in production. The objective is to ensure maximum productivity and performance reliability

In High-Performance Cutting (HPC), cutting tools have to stand their ground whilst simultaneously putting up with quite a lot. Although pure powerhouses can achieve the necessary chip volumes, they often affect the tool's cutting edges, the spindles and the workpiece surfaces. Users do significantly better with robust precision cutting tools that also provide a generous dose of intelligence with their design, statical and dynamically strength and a long life time. In addition to perfect run-out, other vital requirements are strong rigidity and vibration dampening of the cutting tool hat is as pronounced as possible. This makes it possible to achieve large chipping rates and brilliant surfaces with minimal tool wear.

The high feed rates raise the machining forces in HPC, which increases the load on machines tools and cutting tools. It is therefore vital to aim for as much rigidity as possible throughout the system. To avoid damaging vibration during machining, components with a low mass are used and bending arm lengths are kept to a mini-mum. With rigid, light and true running tools,

it is also possible to use conventional machining centers with HPC. Cutting tool materials specially designed for the proce-dure withstand the enormous alternating load on the cutting insert; these are de-signed of carbide, cermet or cubic boron nitride, with coatings and the tool holder is in part, for reinforced with fibers for break resistance.

The main goal of the paper is to present newest research activities dealing with a closed-loop design of coating-substrate-systems, starting with the chip formation simulation around the cutting edge and coming up to new methods for coating-substrate-systems design. The presentation is part of the M3-2S Project.

VIRTUAL TESTING FOR RAPID EVALUATION OF ENGINEERED SURFACE COATINGS

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Significant progress has been made during the past decade in the development of multi-functional surface systems to meet the ever-increasing demands for high performance engineering components operating under severe working conditions. Micro-indentation hardness tests are widely used in the evaluation of candidate coating systems for high load-bearing applications. In a pioneering international collaborative research project, advances in multi-scale simulation techniques and modelling of mechanical damage have demonstrated faithful duplication of test results. This makes it possible to replace mechanical tests with computer simulation, at least as a pre-screening technique to identify, from a wider range of alternatives, shortlist candidate coating systems for particular applications. This reduces the required scope of physical prototype testing, thereby reducing time to market. Furthermore, simulation can both provide insight into the evolution of surface damage, which may suggest mitigation measures and improvements to surface system design. This paper outlines and illustrates the use of a simulation tool for virtual testing of engineered surface systems, highlighting the development of a simple end-user focussed graphical interface that allows application engineers or coatings suppliers to harness the power of sophisticated simulation previously only amenable to researchers and specialists.

ACTIVE SCREEN PLASMA NITRIDING OF AISI M2 HIGH SPEED TOOL STEEL: EFFECT OF TEMPERATURE AND TIME ON MICROSTRUCTURE AND MECHANICAL PROPERTIES

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Active screen plasma nitriding (ASPN) is a novel surface modification technique that has many advantages over the conventional dc plasma nitriding for treating low alloy steels, tool steels and other types of steel components. During this work, the effects of plasma nitriding temperature and time on the AISI M2 high speed steel (HSS) were investigated. To optimise the most suitable active screen plasma nitriding conditions for M2 steel, specimens were treated at different temperatures starting from 400 °C to 525 °C and time duration starting from 30 min to 150 min in an atmosphere of 25% N_2, balance H_2 gas mixture at 75 Pa pressure using 10% substrate bias. A phase analysis of the nitrided layers was carried out using X-ray diffraction technique. Chemical composition, especially nitrogen depth profile measurements, were obtained using glow discharge optical emission spectroscopy (GD-OES), whereas microhardness and load bearing capacity tests were performed using a Vickers hardness tester. Scanning electron microscopy (SEM) was employed to observe the nitrided compound and diffusion layers as well as the indented cracks generated during load bearing capacity tests. Compressive residual stresses present in plasma nitrided M2 steel were evaluated using X-ray diffraction $\sin^2\psi$ method. The nitrided region in our study is mainly composed of a diffusion zone, however a compound layer in the form of γ'-Fe_4N and ε-$Fe_{2-3}N$ phases tend to develop with the increase in nitriding temperature. It has been found that

plasma nitrided temperature and time duration can significantly affect the mechanical properties such as hardness, load bearing capacity and residual stresses present in the nitrided layer.

FRACTURE BEHAVIOR OF HARD THIN FILMS BY IN-SITU SEM BENDING TEST - SESSION H1

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Hard thin coatings are playing a very important role in surface engineering. Nevertheless, limited studies on their failure mechanisms in different loading conditions were reported. This is mainly due to the challenges in determining the mechanical properties of thin coatings.
In the present paper a study on the fracture mechanisms of mono (CrN and ZrN) and multilayer (CrAlSiN) coatings deposited on different heat treated steel substrates in different loading conditions is reported. Coating deposition was performed with an industrial Cathodic Arc Evaporation PVD system. Two different techniques using different loading modes were applied to study the fracture behaviour of such coatings: 1) evaluation of load bearing capacity (LBC) with ball indenter penetration test; 2) in-situ SEM four point bending tests. Fragmentation processes and stress distribution inside the coating were analysed using both LBC and four point bending test. Fracture strength and fracture behaviour using both methodologies were analysed.
The main conclusions achieved are:

- 1. Channel cracking is the failure mechanism for CrN and CrAlSiN;
- 2. Fragmentation occurs in all coatings independently of the loading mode applied;

3. The multilayer coatings exhibit higher fracture resistance than the reference monolayer coatings

STRESS-STRAIN RESPONSE OF HARD THIN FILMS BY MICROPILLAR COMPRESSION

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Compositionally graded and nanostructured thin films not only provide new technological opportunities, but are also interesting from a scientific perspective. Deliberate design of such advanced thin films has revealed superior and unique combinations of mechanical properties, such as high hardness, toughness, wear resistance etc.

In this study, we focused our attention on the evaluation of the residual stress of the deposited coatings as well as determining the mechanical response of "only" the coating thus without the influence of the substrate. Since the structure and thus the mechanical properties of the film are influenced by the substrate, the films under study must be deposited on the substrate which will be used in the technological application. In particular, the material systems under investigation are mono (TiN and CrN) and nano-multilayer (CrN/TiN) coatings deposited on heat treated AISI M2 substrates. Coating deposition was performed with an industrial cathodic arc evaporation PVD system.

1. The residual stress is determined by cutting out a beam (film thickness x 5um x 30 um) composed only by the coating material from the sample using a focused ion beam technique (FIB). The curvature of the freed beam allows to determine the residual stress in the film using Stoney's equation. The stress-strain curve as well as the fracture stress of the film is determined using micropillar compression experiments. The micropillars are fabricated by FIB on the sample cross-section and

thus the pillar is composed only of the coating material and sits on the coating material avoiding the influence of the more compliant substrate material.

A COMPARATIVE STUDY OF THE MICROSTRUCTURE AND TRIBOLOGICAL PROPERTIES OF NANO-MULTILAYER TIALSIN AND CRALSIN COATING SYSTEMS

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Nano-multilayer coatings are drawing more and more attention due to their attractive combination of high hardness and toughness. In this work, nano-multilayer TiAlSiN and CrAlSiN coatings were deposited on AISI M2 high speed steel substrates by cathodic arc evaporation (CAE) deposition in nitrogen atmosphere with the target of powder metallurgy AlSi and pure Ti or Cr. The optimised pressure, bias and current of targets based on previous research were used with rotation speeds of the sample holder varying from 2 to 10 rpm.

Detailed microstructural characterisation of these nano-multilayer coatings was conducted using XRD, SEM, TEM and glow discharge optical emission spectroscopy techniques. Residual stresses present in the coatings were evaluated using X-ray diffraction $\sin^2\psi$ method. Microhardness and load bearing capacity (LBC) tests were performed using a Vickers hardness tester and the adhesion between the coating and the substrate were evaluated by scratch tests. Rotating-sliding wear tests against a WC ball were conducted on selected samples at the room temperature and 400℃. The experimental results show that the bilayer thickness and the phases present in the coating depend strongly on the sample holder rotation speed during the deposition process and the best performance were found for nano-multilayer samples deposited with the rotation speed of 10 rpm. The CrAlSiN coatings outperformed the TiAlSiN coatings in terms of higher resistance to scratch, oxidation, and wear.

COATED SURFACES IN STATIC LINE CONTACT: NUMERICAL SIMULATION AND EXPERIMENTAL VALIDATION

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Load bearing capacity (LBC) is becoming a demanding parameters for high strength components, especially in machine elements. Here, the request for high power density is continuously increasing due to the introduction of small powerful engines. As a consequence the severity of mating surface interaction grows, putting great demands on surface modification processes. Nevertheless, in such field trial and error approaches are still dominating with few experiences on numerical simulation are present.
The aim of this paper is to analyze the case of static line contact between two cylinders bodies, one of them being surface modified with mono and multilayered systems. Actually, line contact is representative of many bearing elements such as gears and rolling bearing components.

The studied surface modified systems are CrN, TiN and TiN/CrN nano-multilayer coatings applied on 42CrMo4 steel substrates. Part of the steel substrates was previous to coating subjected to active-screen plasma nitriding, whereas the other part was in simple quenched and tempered state. Coating deposition was performed with an industrial Cathodic Arc Evaporation PVD system.

A FE Model developed for the analysis of failures of multi-layers coated surfaces subjected to the indentation is extended to the surface-damage analysis for the case of static line contact. This model featured with parameterized and cohesive-zone approach, allows for the propagation of cracks in the coating-layer being simulated, in parallel, critical load inducing the first crack under the varied situations being determined accurately and efficiently. The submodelling technique enable the scalling-down of geometrical model from macro-scale to micro-/nano-scale in order to study the fine cracks in the thin coating layer.

It was observed, for all configurations, when one cylinder is pressed against the other, the crack occurs at the contact edge of coated surface due to the bending effect. Comparison with experimentally derived data are also reported and discussed. To validate the numerical model static contact test of two roller specimens was used. Different levels of loads were applied on the modified surfaces, being the critical load experimental definition given by the load at which surface cracks nucleate. Cracks were detected with scanning electron microscopy observations.

The main achievements of the research are:

2. the CrN/TiN nano-multilayer coating outperformed the monolayer TiN or CrN coating
3. duplex treatment combining plasma nitriding with PVD coating possesses a much higher LBC than single coating

good agreement between modelling and experimental results has been achieved.

POSTER PRESENTATIONS

P-1

MODIFYING OF REX 734 STEEL PROPERTIES IN THE PROCESS OF GLOW DISCHARGE ASSISTED NITRIDING COMBINED WITH PRODUCING OF CARBON LAYER

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Austenitic steels, due to their unique properties, like plasticity, paramagnetism and good corrosion resistance, are becoming more and more widely applied in the chemical and food industries as well as in medicine. Austenitic steel REX 734 is particularly used in medicine for bone implants like hip joint endoprosthesis and medical instruments. It is essential to improve properties of this alloy, and especially its insufficient corrosion resistance in aggressive corrosive environments and very poor frictional wear resistance. Application of glow discharge assisted processing of REX 734 steel combined with production of carbon layers in one technological process enables to modify functional properties of this steel.
This research work included application of low-temperature glow discharge assisted nitriding (_380 °C) combined with producing of carbon layer on REX 734 austenitic steel.
Examinations of morphology and topography of produced layers were made as well as testing of roughness of their surface. Examinations of chemical composition were made with the use of energy dispersive X-ray spectroscopy and examinations of phase composition were made with the use of X-ray diffractometer while Raman spectroscopy was applied for conducting examinations of carbon layer structure. Testing of functional properties of produced layers were also made, such as hardness and frictional wear resistance as well as corrosion resistance with the use of potentiodynamic method.
Obtained results show a considerable influence of an application of hybrid processes on the properties and microstructure of REX 734 steel.

Keywords: carbon layer, austenitic stainless steel, wear resistance, corrosion resistance, glow discharge plasma assisted treatments

P-2

EVALUATION OF DIFFUSION LAYER OF NITROGEN PLASMA NITRIDED TOOL STEELS

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Plasma nitriding is a unique candidate for treating high precision components for a higher surface hardness that maintains the surface conditions and the material's core properties. However, most traditional nitriding methods produce a brittle compound layer on top of the diffusion layer and the costly cleaning or grinding to remove the brittle white layer is required. Thus, plasma nitriding due to diffusion of nitrogen neutral particles is desirable as it can produce a hard wear resistant surface without brittleness and galling that may occur due to the formation of the iron nitride compound layer.

In this research, the bright nitriding of SKD61 tool steel was carried out by neutral nitrogen species in electron beam excited plasma. the electron beam excited plasma (EBEP) source is used as the controllability of the system makes it superior candidate for plasma nitriding. To repel all positive ions, the bias voltage of sample is controlled to positive more than the plasma potential.

The results of our experiments show that the surface of neutral nitrided sample had mirror finishing similar to untreated and only the diffusion layer were generated. The measured hardness and depth of the diffusion layer of all tool steel work pieces surfaces were increased by more than two times that of the core material.

P-3
PLASMA NITRIDING AND NITROCARBURIZING OF A SUPERMARTENSITIC STAINLESS STEEL

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The supermartensitic stainless steels (SMSS) exhibit mechanical properties much better when compared to conventional stainless steels. These steels are widely used in components of equipments in chemical, petrochemical and oil industries, due to its good ductility combined with its lower cost. Several works have shown that plasma nitriding and nitrocarburizing at low temperatures produces a hard surface layer which results in increased wear resistance. On this work plasma nitriding and nitrocarburizing were conducted in a vacuum chamber at 400, 450 and 500°C with a pressure of 5mbar, for 5h in direct current mode. The employed gas mixture was 80% H_2 and 20% N_2 for nitriding and 80% H_2, 17% N_2 and 3% CH_4 for nitrocarburizing. The plasma treated SMSS steel samples were characterized by means of optical microscopy, scanning electron microscopy, Vickers microhardness, X-ray diffraction and wear tests. It was found that increasing plasma nitriding or nitrocarburizing temperature, from 400 to 500°C, the thickness of the layer increases. X-ray diffraction patterns of the plasma treated samples presented wider peaks dislocated to the left (higher d values) relative to the diffraction patterns of a standard martensitic matrix, which is probably due to a lattice constant expansion caused by the nitrogen insertion. The worn volume lost after plasma treating decreases for all temperatures.

P-4
THE NUMERICAL SIMULATION AND PREDICTION OF MICROSTRUCTURES AND MECHANICAL PROPERTIES OF THE WHEELS SUBJECTED TO QUENCHING PROCESS

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The FEM simulation of temperature and transformation of super-cooled austenite in the wheel has been performed during its spraying quenching process, and the final microstructures and mechanical properties have been predicted. The cooling curves on some points of the wheel were obtained through the thermocouples placed in wheel subjected to spraying quenching. The heat transfer coefficient between wheel surface and spraying water was calculated by inverse heat conduction method. The isothermal austenite decomposition experiments have been performed under different temperature to get the pearlite with different lamellar spacing characterized by SEM, and mechanical tests have also been carried out to determine the mechanical properties such as tensile strength, yield strength and impact toughness. The relations between transformation temperature, lamellar spacing of pearlite and mechanical properties was thus established. The introduction of these relations in quenching simulation makes it possible to predict the final mechanical properties in the quenched wheel. Validation experiments show that the simulation results fit well with those tested in practical manufacture. The present study provides solid basis for quenching process optimization and ensure the products quality.

P-5
ANALYSIS AND NUMERICAL MODELLING OF CERAMIC PIEZOELECTRIC BEAM BEHAVIOR UNDER THE EFFECT OF EXTERNAL SOLICITATIONS

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The piezoelectric materials have become indispensable in many technological applications. Nowadays, they are present in many applications fields such as in applied mechanics, aeronautics, biomechanics and civil engineering in order to control their behavior and predict their life span. These materials have an inverse piezoelectric effect which allows them to control the form and to present any neither noise nor vibration at any time or position on the structure. In this study we are interested in the bending behavior analysis and modelling of a ceramic beam under external solicitations using numerical simulations based on the finite element methods. The modelling permit to simulate the deformations in a piezoelectric ceramic beam subjected to an electric field and to simple mechanical stress taking into account the electromechanical coupling. It has been found that the obtained analytical results are in a very good agreement with those obtained by numerical modeling. As a result, the interest of such modelling analysis allows the design, the conception and the optimization of mechanical systems based on piezoelectric elements. These latter, known as smart or "intelligent" materials, are often used to measure and / or to control finite deformations or vibrations of the mechanical systems, so that to prevent their plastic deformations or their total failure.

Keywords: Piezoelectric material, ceramic beam, intelligent structure, finite elements modelling**.**

P-6
EFFECT OF NANO-PARTICLE ADDITIVES ON ATRUCTURE AND WEAR PERFORMANCE OF MICRO-ARC OXIDATION COATING PREPARED ON 2A12 ALUMINUM ALLOY

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Nano-partcle reinforced composite coatings were prepared on 2A12 aluminum alloy by micro-arc oxidation and the effect of SiO_2 nano particles on structure and wear performance of coating was studied. By means of SEM, CETR micro-nano-indenter friction and wear tester and white-light interferometer, the surface morphology and the friction and wear property were tested. Results showed that compared with the common coating without nano-particle

addition, the size and amount of nano-particle reinforced composite coatings with 20nm SiO_2, 80nm SiO_2 were decreased substantially, and the wear resistance of the micro-arc oxidation coating was improved. While in case of 800nm SiO_2 addition, the porosity was not enhanced, and the wear performance of coating was not improved.

P-7
CORROSION RESISTANCE OF PLASMA NITRIDED AND NITROCARBURIZED AISI 316L AUSTENITIC STAINLESS STEEL

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The usage of coatings in surface engineered components is increasing due to the need for improved hardness, corrosion and wear resistance. Austenitic stainless steels are the most important family of stainless steels with respect to both the number and types of applications. Plasma nitriding and nitrocarburizing of that steels can produce layers composed by a phase called expanded austenite, "S-phase or g_N". This interesting phase is supersaturated with respect to nitrogen and is characterized by high hardness and wear resistance. On this study plasma nitriding and nitrocarburizing of AISI 316L steel were conducted in a vacuum chamber at 400, 450 and 500°C with a pressure of 5mbar, for 5h in direct current mode. The employed gas mixture was 80% H_2 and 20% N_2 for nitriding and 77% H_2, 20% N_2 and 3% CH_4 for nitrocarburizing. The plasma treated AISI 316L steel samples were characterized by means of optical microscopy and Vickers micro-hardness and corrosion tests. The corrosion characterization was carried out by obtaining the potentiodynamic polarization curves in artificial sea water (NaCl 3,5%). After the plasma treatments it was observed that layer thickness increases after increasing temperature, from 400 to 500°C. As carbon diffuses faster in austenite than nitrogen, the addition of methane on the treating gas mixture makes the nitrocarburized case thicker than the nitrided. The treatment at 400°C produced a homogenous S-phase layer while at 450 and 500°C the X-ray diffraction indicate the presence of chromium and iron nitrides and/or carbides. The potentiodynamic polarization curves show that resistance against corrosion is higher for the samples treated at 400°C, when comparing to the untreated substrate. A change on the dominant corrosion mechanism was also observed after nitriding or nitrocarburizing, from localized pitting corrosion to general corrosion.

P-8
DECOMPOSITION OF EXPANDED AUSTENITE IN AISI 316L STAINLESS STEEL NITRIDED AT 450°C

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Expanded Austenite can be produced during plasma nitriding of austenitic stainless steels and it has high levels of strength, toughness and corrosion resistance if compared to the traditional layers composed by nitrides. However, the expanded austenite properties are significantly harmed with its decomposition, caused by its thermodynamic metaestability. In the present work samples of AISI 316L austenitic stainless steel were investigated after plasma nitriding at 450°C for 5 hours under pressure of 5mbar. Optical microscopy has showed the formation of a nitrided layer with homogeneous microstructure and thickness. The X-ray diffraction analysis (XRD) has been able to confirm the presence of expanded austenite and its typical anomalous behavior. The FCC lattice parameter increment measured from {200} reflection is significantly higher than those estimated from another diffracted peaks. The observation of nitrided layer by transmission electron microscopy (TEM) has allowed

identifying some dispersion of fine rounded particles, with sizes between 10 and 15nm. Selected area electron diffraction (SAED) analyses indicate that these particles are chromium nitrides with some preferential orientation (texture) with the matrix. The lattice parameter of expanded austenite calculated by SAED is slightly larger than that determined by XRD, due to the effect of the removal of substrate from TEM sample. During TEM observations have been occasionally identified some areas on the samples with a singular lamellar morphology, very similar to the pearlite colonies found in carbon- steels. SAED analysis suggests that these areas are composed by BCC ferrite and cubic chromium nitrides, produced after a local decomposition of the expanded austenite layer. The occurrence of the expanded austenite decomposition can be related to the microsegregation of chromium and molybdenum in the nitrided layer, therefore EDS microanalysis have confirmed increased amounts of these elements in relation to the not decomposed areas.

P-9
EVALUATION OF AN INTENSIVE QUENCHING SYSTEM THROUGH THE HEAT TRANSFER CHARACTERIZATION

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Lately, due to environmental and security challenges, there is a great interest in finding alternatives quenchants for replacing mineral oil. The intensive quenching process uses water as cooling medium. This process seems to be capable to promote a substantial improvement in mechanical properties, with potential reduction of distortion and cracking of metal components. Furthermore it allows reducing or eliminating the time of carburizing, as the use of low alloy steels. This work aims to evaluate if a laboratorial system that has been built for this purpose can be considered an intensive quenching process. The analysis will be performed by means of cooling curves obtained from stainless steel probes, from which it be calculated the Biot number, heat transfer coefficients, H Grossman values and thermal residual stresses.

P-10
PHASE TRANSFORMATION AND MICROSTRUCTURED OF NANO-STRUCTURED NI/AL_2O_3 COATING DEPOSITED BY PLASMA SPRAYING

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Nanostructured Ni/Al_2O_3 feedstock was prepared by chemical wrapping, spraying and gas protected sintering, and nano-structured coatings were deposited subsequently by plasma spraying. The feedstocks are loose and porous, and the size are about 20-50um. XRD, SEM and TEM were used to detect the phase and microsture of the coatings. The great mass of α-Al_2O_3 was transferred into γ- Al_2O_3 during plasma spraying process. The Ni/Al_2O_3 coatings are composed of irregular fully-deformed layers. The grain size of the coatings is about 100nm. There are two states of Ni in the coatings, some separate Al_2O_3 phase and the others form sphericity particle enveloped in Al_2O_3.

P-11
SURFACE PROPERTIES AND ECONOMY OF OXIDE COATINGS ON STEEL SHEETS BY THERMAL AND CHEMICAL PROCESSES

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Atmospheric corrosion of carbon steel sheet may be controlled by stabilizing / thickening of the thin oxide film on the metal by thermal and chemical processes. Amongst the processes for oxide conversion coatings on steel is the blueing process which is performed by several techniques. In this research, a comparison has been made between thermal blueing and chemical processes to obtain oxide coatings on carbon steel sheet. Thermal oxidation cycles were carried out on low carbon and high carbon steel specimens and time, temperature and cooling rate were studied as the process parameters. Chemical oxide coatings were produced by immersion of specimens in $NaOH\text{-}KNO_3$ solution at appropriate temperature and concentration, and, the coatings were evaluated according to thickness and surface characteristics. Electrochemical impedance spectroscopy (EIS) was employed to compare the corrosion behaviour of the specimens and the oxide films were optimized in terms of surface colour and EIS characteristics. The structure and morphology were examined by optical and scanning electron microscopy and surface topography was studied by atomic force microscopy (AFM). Experimental results indicate that corrosion resistance of the coated sheets depends on the oxidation process parameters which may be optimized according to the application. The chemical techniques produced thicker coatings and higher corrosion resistance, but, these methods increased the product cost by more than 15%. Surface colour of the oxide is determined by interference effect, being a function of film thickness. But, the corrosion behaviour was found to be independent of the surface colour.

P-12
EFFECT OF ENHANCEMENT OF ATOMIC NITROGEN SPECIES VIA DOUBLE-FOLDED SCREEN ELECTRODE

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As clean and energy saving surface hardening technology, plasma nitriding techniques have been evolved with object of higher performance in the last decades. Even though the diffusion of nitrogen inward to steel is occurred at the final step, solid diffusion from surface, energy transition from gas molecule of nitrogen to atomic or an activated state have many different steps depending on the plasma conditions, parameters and the design of each equipment. And this study made comparative on the nitrogen transfer with conventional DC plasma nitriding process and novel nitriding (atomic nitriding; ATONA) process using plasma diagnosis and metallurgical observation. With different vacuum pressure, gas ratio and new designed electrode, it showed different behavior of plasma nitriding with the nano-sized nitride on the outer surface of steel due to different rate determining plasma species. It was able to identify it by optical emission spectroscopy (OES) studies. From these observations, we could understand better role of ions or neutral nitrogen species, like neutral nitrogen (N), N_2^+ and NH_x radicals in plasma nitriding process with different parameters.

Keyword: atomic nitrogen, neutral nitrogen, diffusion rate in steel, hollow cathode discharge (HCD) effect, plasma species in plasma nitriding process, optical emission spectroscopy (OES).

P-13
TANTALUM CARBIDE DISPERSED HIGH CR-NI CAST IRON PRODUCED BY PLASMA SPRAYING

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The spray material is heated, melted, and accelerated by a high temperature flame in the plasma spraying process. Low-pressure plasma spraying can produce rapidly solidified thick materials because alloy droplets accumulate successively on the substrate, and solidify at a

cooling rate in the range of 10^5-10^8 Ks^{-1}. Depending on the cooling conditions of the substrate and on the alloy composition, deposits can be produced as metastable phases or extremely fine crystalline phases. Plasma spraying is an attractive method for the production of deposits with in-situ formed fine particles. In recent years, much attention has been paid to high Cr-Ni cast iron with vanadium carbide, which is attractive for use in metal molds and pump parts, due to its high wear resistance and high corrosion resistance. In the present work, high Cr-Ni cast iron alloy powder is low-pressure plasma sprayed to produce high Cr-Ni cast iron base alloy deposits with finely dispersed tantalum carbide particles. The as-sprayed deposit produced on a non-cooled substrate was composed of γFe, αFe and carbide. The fine precipitates in the as-sprayed deposit were carbide. With increasing heat treatment temperature up to 1273 K, the carbide particles coarsened. The as-sprayed deposit produced on a non-cooled substrate had higher hardness than the heat-treated deposits. The wear resistance of deposit decreased with increasing heat treatment temperature.

P-14
DEVELOPMENT OF A HIGH-PERFORMANCE GAS CARBURIZING FURNACE

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The advantage of gas carburizing is that it can be performed steadily and reproducibly by controlling the atmosphere, thus differing from low-pressure carburizing. However, gas carburizing has the following disadvantages: thermal efficiency is poor, carburizing speed is low, internal oxidation occurs, and emission of CO_2 gas is large compared to low-pressure carburizing. The objective of this study is to develop a high-performance gas carburizing furnace to improve the inherent disadvantages of gas carburizing.

We developed a batch-type gas carburizing furnace whose maximum process capacity is 200 kg/gross; it consists of a pre-chamber and a heating chamber that can be evacuated. The heating chamber consists of high-efficiency heat-insulating materials and vacuum heat-insulating construction. The carburizing was performed using atmospheric gases with high carbon potential (CP) and high carbon transfer coefficient (β) based on the decomposition gases of methanol. The heating-up and the cooling-down with nitrogen gas, the boost cycle with atmospheric gases of high β and high CP, and the diffusion cycle with atmospheric gases of low CO content were carried out to decrease internal oxidation.

The reduction of more than 90% of the CO_2 gas emission was achieved using a small amount of carrier gases in the air-tight heating chamber and the precise control of the atmosphere during carburizing. No seasoning time was required because the air does not enter into either the heating chamber or the pre-chamber when stopping the furnace. The carburizing speed was higher than that of the low-pressure carburizing because of high β and high CP. The internal oxidation of the gas carburizing was decreased more than 50% compared to the conventional gas carburizing by decreasing the oxidation constituents such as CO_2, O_2, and H_2O using nitrogen gas or atmospheric gases with high CP.

P-15
A STUDY OF A CHANGING OF PHYSICAL AND CHEMICAL INTRA-STRUCTURE ON SI-DLC FILM DURING TRIBOLOGICAL TEST

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The silicon-containing Diamond-like Carbon (Si-DLC) film as an low friction coefficient coating has especially treated a different silicon content by plasma-enhanced chemical vapor deposition (PECVD) process at 500 ℃ on nitrided-STD 11 mold steel with tetramethylsilane

(TMS) gas flow rate. The effects of variable silicon content on the Si-DLC films were tested with relative humidity of 5, 30 and 85% using a ball-on-disk tribometer. The wear-tested and original surface of Si-DLC films were analysed for an understanding of physical and chemical characterization, including a changing structure, via Raman spectra and nano hardness test. The results of Raman spectra have inferred a changing intra-structure from dangling bonds. And high silicon containing DLC film have shown increasing carbon peak ratio (ID/IG) values and G-peak values. In particular, the tribological tested surface of Si-DLC was shown the increasing hardness value in proportional to TMS gas flow rate. Therefore, at same time, the structure of the Si-DLC film was changed to a different intra-structure and increased hardness film with mechanical shear force and chemical reaction.

Keywords: Si-DLC, high temperature synthesis DLC, tribo-chemical reaction, hardness increasing on tribometer.

P-16
INFLUENCE OF DEEP CRYOGENIC TREATMENT ON CUTTING EDGE LIFE IN CUTTING TOOLS

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Resistance to service wear and the life of the cutting edge belong to the most important quality parameters of cutting tools. Particularly for automatic machining centres the changeover of tools means a significant time loss and an obstacle to production continuity. This is why there is a great focus on the tool life optimisation. Achieving optimum life requires, in addition to using special PVD and CVD coatings and appropriate lubrication, a proper heat treatment process. In recent years, deep cryogenic treatment became widely used, for high-alloyed tool steels in particular. This process consists in cooling a material and holding it at temperatures below -60°C. Although some theoretical aspects of this process are still under research in many laboratories, the positive impact of deep cryogenic treatment on the wear resistance of tool steels has been demonstrated in numerous industrial applications.

The present paper describes a method of testing of life of cutting tools developed by the company PILSEN TOOLS s.r.o. in collaboration with COMTES FHT. This method was used for testing turning tools from CSN 419857 high speed steel. Prior to testing, these tools have been heat treated by various methods including conventional hardening with triple tempering and various schedules with a deep cryogenic treatment. Several temperatures and holding times were used in these deep cryogenic treatment schedules. Results of tests indicate that the selection of heat treatment method can have a decisive impact on the cutting edge life. They also show that deep cryogenic treatment leads to a considerable enhancement of the cutting edge life in all cases.

P-17
PREPARATION OF HOMOGENEOUS FE-AL INTERMETALLIC COMPOUND SHEET FROM FE AND AL FOILS BY SPARK PLASMA SINTERING

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The Fe-Al intermetallic compounds, especially Fe_3Al, are remarkable having about 30% lower density than those for existing high-temperature structural material such as stainless steels or Ni-base super alloys. They are excellent in oxidation and sulphurization resistance as an alternative material. In general, because the intermetallic compound has a high melting point, it is produced from the ingot metallurgy method and powder metallurgy methods. However,

because these methods have a disadvantage such as the nonuniformity of the sample composition, machining such as extrusion or forging and additional processing such as heat treatment for a long time are needed. Spark plasma sintering (SPS) process enables one to sinter materials at lower temperatures and in shorter times than conventional sintering techniques. In this study, Fe-Al intermetallic compound was prepared from elemental Fe and Al foils by SPS process. The microstructures produced at each processing stage were characterized by optical microscopy and scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS). Vickers microhardness testing was used for hardness determination. After the SPS process of the first treatment for 3.6 ks at 873 K followed by the second treatment for 3.6 ks at 1373 K, a homogeneous Fe_3Al intermetallic compound was obtained.

P-18
EFFECT OF COOLING RATE AFTER NORMALIZING ON BASE-METAL PROPERTIES FOR POST WELD HEAT TREATED PRESSURE VESSEL STEELS

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Post Weld Heat Treatment (PWHT) is commonly used in the pressure vessel construction to reduce the residual stresses on welded regions formed during welding. However, the properties of base-metal decreases after long-term thermal aging especially in normalized base-metals. In the present study, the effect of cooling rate after normalizing on microstructure, mechanical properties and hydrogen assisted cracking property after PWHT were investigated. Two kinds of steels were fabricated by 15℃/s, 10℃/s, 3.5℃/s, 0.5℃/s cooling rate. PWHT was performed at 620℃ for 3h. Charpy impact test and tensile test were conducted before and after PWHT. Microstructures were observed using Optical Microscope, Scanning Electron Microscope and Transmission Electron Microscope. HIC (Hydrogen Induced Cracking) test was also performed. After HIC teat, HIC sensitivity was measured by ultrasonic detection. From the results of Charpy impact test, the toughness of base-metal was improved as increasing cooling rate of normalizing. The HIC test results indicated that all specimens after PWHT showed excellent HIC resistance. But the properties of HIC before PWHT depended on chemical compositions of steels.

P-19
TREATMENT BY VIBRATIONS FOR GRANULAR WORK-PIECES

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Actually, without increase in productivity constant and improvement of products quality are difficult to get a development in mechanical engineering field. These preventions can be avoided by introducing new processes which include a surface treatment by vibration to result a superficial plastic deformation for granular work-pieces. Recently, these procedures are useful and reliable in different stages of industries when high surface quality requirements. Present work focused and based on an experimental model can be used for strengthening and in surface finishing process for granular work-pieces, which that enhance the quality of the surface layer for the test pieces. The resulting model allows representing each index as a function of initial data setting, which are: material properties and controlling process parameters. These relationships give as an initial step to determine the optimum process parameters. During production series, the surface finishing process and strengthening treatment for granular test pieces are presented, about calculations results also approbated industrially.

P-20
CLEANING PRIOR TO PVD/CVD-COATING:DESCRIPTION OF CLEANING PROCESSES DEPENDING ON CONTAMINATION AND MATERIAL OF SUBSTRATES, DETERMINATION OF THE CLEANLINESS

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Cleaning becomes more and more an industrial operation like turning, milling or coating. The importance of mechanical parts cleaning in industry however discloses its importance to many manufacturers only when particular contaminations lead to the failure of the component and finally - in the worst case - requires expensive callbacks. Therefore it is essential to include mechanical parts cleaning as a value adding operation into the manufacturing.

The presentation covers the principles of aqueous cleaning in either spray wash machines or ultrasonic systems and explains the variable parameter of the process. Further some typical cleaning process will be presented and explained. Methods to attest to the cleanliness of substrates will be presented.

Keywords:

- - Aqueous Cleaning as a value adding process
- - Importance of adequately cleaned substrates in industry
- - Methods to measure the cleanliness

P-21
SYSTEMATICAL ANALYSIS OF COOLING CURVES FOR LIQUID QUENCHANTS

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This paper suggests and describes a systematic approach to the analysis of liquid quenchant characteristics using a standard ISO Inconel alloy probe. A new systematic mathematical model for the cooling curve analysis has been developed. The model allowes a possibility for a more precise determination of quenching parametars and links certain parametars with a mathematical model for microstructure prediction and properties of quenched work pieces. Besides the usual technological parametars (cooling rate at 700^{o}C, maximum cooling rate, cooling time from 800 to 500 ^{0}C, cooling rate at 300 ^{0}C, quenching intensity H), heat transfer coefficient is determined using the following methods: lumped-heat capacity method, Kobasko method, inverse heat transfer method according to "SQIntegra" software and new asymptotical-inverse heat transfer method. The results of heat transfer coefficient calculation according to all the mentioned methods are compared and verified with ISO Inconel alloy probe cooling curve analysis for quenching in water, industrial quenching oil and water based PAG polymer solution with concentrations from 5 to 30%vol PAG in water.

P-22
THE EFFECT OF AUSTEMPERING TEMPERATURE ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF NI-MO GRAY CAST IRON

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Austempering, as a proper heat treatment process for providing desired characteristics through forming bainite Phase in Cast Iron pieces, has always been a matter of concern for researchers and industrialists. In this study, a considerable number of wedge-shaped samples have been manufactured by casting. Then by using heat treatment process the effect of various austempering temperature at a constant austenitising temperature of 900 ℃. In order to so, on the structure and also mechanical properties of the samples have been examined. First, the samples were austinited in this temperature and afterwards rapidly conveyed to molten salt bath with different temperatures of 380, 400, and 420 ℃. The results obtained from metallography method, processed images of optical microscope and also mechanical tests on existing samples admit that as the austempering temperature increases the percentage of bainite phase in the structure of wedge-shaped samples structures ascends and this obtained structure improves the mechanical properties as well.

P-23
THE EFFECT OF COOLING RATE ON MICROSTRUCTURE OF AUSTEMPERED NI-MO GRAY CAST IRON

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Taking into account the importance of the amount of bainite phase on the microstructure of cast irons and its influence on the improvement of mechanical properties, this research selected an alloy of gray cast iron containing Nickel-Molybdenum and conducted the austenitising and austempering processes at 900 ℃ and 400℃ for 60 minutes respectively. The way of bainite phase formation and the effect of sample thickness, i.e. cooling rate, were examined by selecting a standard staircase sample. The results indicated that, by increasing the cross sections of samples, the martensite percentage decreases and the phase proportion of bainitic ferrite increases.

P-24
INTERNAL OXIDATION DURING STEELS CARBURISING

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Causing the decreasing of the surface hardness and of the mechanical characteristics as strength, plasticity, toughness and especially fatigue, the phenomenon of internal oxidation occurred during carburising of steels alloyed with chromium, titanium, silicon, manganese etc request a particular attention. In the presence of alloying elements with high affinity for the oxygen dissolved in the metallic matrix and for a diffusion coefficient of oxygen along austenitic grain boundaries increased by several orders of magnitude due to vacancies in excess, the premises for the appearance in these areas of certain oxide films with intergranular disposal were created. This phenomenon occurs more intensively in the case of atmospheres with high carbon monoxide, produced directly in workspace of heat treatment equipment, as is the case of the atmospheres produced by instillation and pyrolysis of organic liquids.
The paper reviewed the general aspects of the phenomenon of internal oxidation during carburizing of alloy steels in gaseous media, with particularization for carburising media obtained through instillation and pyrolysis of organic liquids.

P-25
HEAT TREATING OF HOT ROLLED STEEL RINGS

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A series of tests were conducted on hot rolled steel ring to evaluate their heat treatment conditions and determine the transformation kinetics. The rings were made of medium alloy carbon steel of a composition close to that of AISI 4140 and were instrumented by placing up to 20 themocouples in five diferent sectors. The rings were solubilized following a stepwise cycle that lasted for 12 hours, the last four hours at 880°C, and were left to cool in air or inmersed into a quenching bath. The onset of the various transformation reactions that take place during heating or cooling were obtained by thermal analyses of the temperature-time curves. An algorithm was designed to derivate the curves; the critical points were deduced from the inflections detected when the time derivative was plotted as a function of either time and temperature, as first order reactions are either endothermic or exothermic and affect the cooling or heating rates. The critical temperatures during cooling were used to draw a continuous cooling transformation diagram. Metallographic observations carried out on the sections confirmed the CCT diagram.

P-26
OXIDATION OF SI AND SI-CU BEARING STEELS

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A series of samples of silicon and silicon and copper bearing steels were oxidized in air within the temperature range of 950 to 1150°C for times of 5 to 20 minuters. The samples were reheated with the aid of an induction furnace to reduce oxidation during the heating cycle and were cooled to room temperature with forced air. The intervals of time and temperature are consistent with those to which steel slabs and blooms are subjected to during the intermediate roughing stages in modern mills. The oxide crusts were examined by optical and scanning electron microscopy. It was found that the oxide crust in the silicon steel was made of an internal layer of fayalite ($FeO\text{-}SiO_2$) and an external one of wustite (Fe0), whereas that of the Si-Cu bearing steel was made of the commonly encountered layers of hematite (Fe_2O_3), magnetite (Fe_3O_4) and wustite, with a thin layer of fayalite close to the steel interface. The crust was thinner in the Si steel in all cases; internal oxidation of Si was found to occur in this steel. A copper rich layer was found in the Si-Cu bearing steel heated at 1100°C, which is due to its low melting point and to its resistance to oxidation at this temperature; enrichment was not found to occur at 1150°C. It was found that internal oxidation of silicon occurred at all temperatures, and this is the reason for the rolled-in scale that is observed in silicon containing steel and its difficulty to eliminate by pickling.

P-27
A FINITE ELEMENT ANALYSIS FOR VACUUM CARBURIZING-HEAT TREATMENT OF AN AUTOMOTIVE GEAR RING

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A finite element model was formulated to simulate the carburizing, temperature, phase transformation and deformation behavior of an automotive gear ring during vacuum carburizing-heat treatment process. First, a carbon concentration profile of gear ring was

calculated by a finite element analysis considering the heat transfer and diffusion equation. Based on the results of carburizing analysis, in turn, the finite element analysis, which could consider transformation induced plasticity during phase transformations, was performed to understand the effect of carbon concentration on phase transformation and deformation behavior during the heat treatment. The analysis verifies that carbon concentration play significant roles in strengthening by transforming hard martensitic phase.

P-28
COMPARISON OF THE QUENCHING AND HEAT TRANSFER PROPERTIES OF AN EXPANDED SERIES OF VEGETABLE OILS

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Vegetable oils may be potentially used as an alternative basestocks to petroleum oil for the formulation of quenchants. Comparative cooling curve performance of a number of vegetable oil basestocks which are commercially available in Brazil was reported previously. These basestocks included: soybean, canola, corn, cottonseed and sunflower oils. However, these particular vegetable oils exhibit very poor thermal-oxidative stability relative to petroleum oil which is attributable to its nature of the molecular composition. To address this specific deficiency which affects long-term performance in use, a number of additional vegetable oils with much more favorable molecular structure were examined including: peanuit (groundnut) oil, palm oil and coconut oil. To assess the quenching performance compared to conventional and accelerated quenching oils, cooling curve performance according to ASTM D6200 for a series of increasing bath temperatures was determined. In addition, the heat transfer coefficients were calculated and compared. It is noteworthy that the vegetable oils did not exhibit classical film boiling or nucleate boiling behavior during quenching. The results of these studies will be reported in this paper.

P-29
OXIDATIVE STABILIZATION OF VEGETABLE OILS BY STRUCTURAL MODIFICATION AND HEAT TRANSFER PERFORMANCE DURING QUENCHING

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Vegetable and animal oils as a class of fluids have been used for hundreds of years, if not longer, as quenchants for hardening steel. However, when petroleum oils became available in the late 1800s and early 1900s, the use of these fluids as quenchants, in addition to their use in other industrial oil applications quickly dimished. This was primarily, but not exclusively, due to their generally very poor thermal-oxidative instability and the difficulty for formulating fluid analogs with varying viscosity properties. Interest in the use of renewable fluids, such as vegetable oils, has increased dramatically in recent years as alternatives to the use of relatively non-biodegradable and toxic petroleum oils. The relatively poor thermal-oxidative stability has continued to be a significant reason for their general non-acceptance in the marketplace. Soybean oil is one of the most highly produced vegetable oils in Brazil. Currently, there are commercially produced epoxidized versions of soybean oil which are available. The objective of this paper is to discuss recently obtained results showing the dramatic improvement in thermal-oxidative stability of epoxidized soybean oils and to discuss their potential use and heat transfer properties as viable alternatives to petroleum oils for hardening steel.

P-30
USING THE MINERAL FEED OF REPUBLIC OF KAZAKHSTAN IN FABRICATION OF FIREBRICKS OF ALUMOTHERMITE FUSION WELDING OF RAILWAY JOINTS

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Rails fused by alumothermite welding, made of new and utilized rails, thermally not strengthened, of open-hearth and Bessemer origin, are devoted to be railed on main and station arriving and other ways, point works including.

Data about joint holes dimensions and their placing depending on the rail types are stated in GOSTs (State Standards). Nowadays the special refractory bricks are used in rail alumothermite fusion welding of railways' units of point works. Refractory crucibles when melting metallic powder at 1700℃ is used also in point works. Alumothermite welding of railway joints carry some difficulties, connected with thin, hard and infusible surface of aluminum oxide AL_2O_3, causing gas cracks on the surface. The film of oxide covers drops of melted metal and puts obstacles in the way of fusing with each other and main metal. To crash and taking off the film and metal protection from repeated oxidation when welding, they use special flux metal or welding is carried in atmosphere of inactive gases. Fluxes consist of alkali-earth chloride salts (NaCl, KCl, BaCl, LiF_2, CaF_2). The welding is done by melting electrode at DC of reverse polarity and welding by non-melting electrode at AC.Aluminum and its alloys also are welded either by electro slag welding or is good in contact welding. Thermite appears to be a powder like mixture of metal oxides together with deoxidants. At definite temperature heat it occur chemical reaction between thermite mixture components, followed by significant quantity of heat. That heat is used to warm up the welding parts to yielded or liquid condition. Thermite welding performed by thermite looking like a powder mixture of aluminum and irodross. However the much heat is evolved and the temperature rises to 1700℃.

Key words: alumothermite welding, railway joints, mixture components, significant quantity, deoxidants, materials.

P-31
SURFACE HARDENING OF STEELS BY LASER ALLOYING AND NITRIDING

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The article deals with improving the strength of steel by surface treatment by laser heating and nitriding.

Developed two-stage technology of surface hardening of carbon steels. At the first stage steels were subjected to laser alloying nitride-forming elements, and at the second stage nitriding is carried out.

Established technological regimes of laser treatment, at which the steels formed homogeneous in composition superfine-grained ferritic-pearlitic structure, and in the high-carbon steels there are finely dispersed carbides.

The disadvantage of laser treatment are the high tensile stresses at the zone of laser treatment that reduce the characteristics of crack.Therefore, the heating in the nitridation has positive effects on these properties.Simultaneously, the nitriding formed finely dispersed

coherent nitrides of alloying elements that increase the hardness of the surface layer up to 20000 MPa.

It is established that such a two-stage treatment improves the wear resistance of steels in the 3-15 times, and the crack-resistance in 1,5 times.

P-32
CRITICAL HEAT FLUX DENSITIES AND THEIR IMPACT ON DISTORTION OF GEARS DURING BATCH QUENCHING (PART 2)

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The paper discusses double distortion during batch quenching of gears caused by local film boiling. The present work is the second part of the authors' investigations. The first part was widely discussed at the WSEAS Conference held on August 20-22, 2010 in Taipei, Taiwan. On the basis of experiments, CFD modeling and FEM calculations of residual stress distribution in gears, the authors came to the conclusion that distortion and variations in dimensional change can be significantly decreased by combining water flow velocity with the special chemical additives which increase the first critical heat flux density. During batch quenching of large gears in an agitated, anticorrosion water-salt solution of optimal concentration, distortion variation is small and dimensional change is repeatable from tooth to tooth. During intensive quenching of small gears in a high velocity IQ system distortion sometimes is not repeatable, especially when local film boiling occurs between gear teeth. Computer simulation shows that double distortion can occur when local film boiling takes place between two adjacent teeth. It means twice bigger distortion when two local film boiling occur between two teeth in two areas located far from each other. In the first area one tooth will move to left side and in the second area similar tooth will move to right side. In this case the distortion will be multiplied by two times. Several measures have been addressed to decrease distortion variation, namely to optimize the concentration of brine solution. There is a need to further investigate critical heat flux densities which have a great effect on distortion of steel parts during quenching. A method and software is being developed to design a standard for critical heat flux density evaluation. It makes sense to combine the efforts of many companies worldwide to start building a database of critical heat flux densities to be widely used in practice to solve distortion problems. A method and software for optimizing the chemical composition of steel have been developed by IQ Technologies Inc. and Intensive Technologies Ltd. for use in decreasing distortion and increasing service life of steel parts.

P-33
CRITICAL FACTORS ON THE ADHESION OF ANODIC TiO_2 NANOTUBES TO TITANIUM SURFACE

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Poor adhesion of TiO_2 nanotubes (TNTs) to the underneath titanium is important since there are many applications for which a good mechanical and electrical contact to the substrate is necessary for an efficient performance. Adhesion of TNTs layer formed in ethylene glycol on commercially pure titanium sheets was investigated in aqueous solutions by considering a new barrier layer formation between nanotubes and titanium substrate which affects their adhesion and properties. Dissolution of titanium fluoride layer between nanotubes and the substrate in aqueous solutions results in detachment of nanotubes from the substrate and formation of a new barrier layer on titanium surface by anodic polarization. We have shown that electrochemical and photo-electrochemical properties of nanotubes after polarization in

aqueous solutions are influenced to a great extent by properties of the newly formed layer. Ethylene glycol (99.8% anhydrous) with addition of 0.25 wt% HF and 0.75 wt% deionized water were used as anodization electrolyte. Potential was kept at 45 V during anodization for different durations to form nanotubes of various lengths. A Field-Emission Scanning Electron Microscope was used to examine the morphology and geometry of nanotube arrays. Re-anodization process was carried out in 0.5 M K_2SO_4 solution up to 5 V using a PARSTAT instrument with Silver/Silver Chloride (SSC) reference electrode at 20 mV/s sweep rate. A platinum mesh was used as counter electrode. Photo-electrochemical characterizations were conducted in a quartz cell using 0.1 M ammonium biborate electrolyte, a platinum wire counter electrode and a SSC reference electrode. The differential capacitance of the growing film was recorded during re-anodization process by means of a two-phase lock-in amplifier. Detachment of TNTs formed in organic solutions from titanium surface while rinsing with water or drying thereafter is not in agreement with good adhesion of TiO_2 barrier layer formed anodically on titanium.

P-34
INFLUENCE OF THE THERMAL SPRAY ON THE 7475 ALUMINUM ALLOY

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Aluminium alloys are extensively used in aircraft industry due to its good strength / weight ratio. However, their wear and corrosion resistances are too low. The possibility of using protective coatings that do not compromise their mechanical strength constitute therefore a highly romising alternative to increase the use of such alloys. In this work, samples of precipitation hardened 7475 aluminum alloy were thermal spray coated with the following coatings: H 1275 (4Fe-16Cr-4Si-3.5B-0.8C-bal Ni) and 1256 F (28Cr-4W-3Fe-3Ni Co-bal). The process did not significantly influenced the hardness of the substrate. Wear tests of the type 'calotest " showed a sharp increase of wear resistance in comparison with the substrate.

P-35
EVALUATION OF WEAR RESISTANCE OF PEEK-BASED COATING

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PEEK (Polyether Ether Ketone) is regarded the one of the highest performing existing thermoplastic materials due its high temperature capability (260^{o}C). It is insoluble in all common solvents and have excellent resistance to acids, bases, hydrocarbons, salts and steam. Its high cost prevents its use as bulk material. However, it can be used as a coating, thus reducing component cost. In this work, 30x25x3mm aluminum samples were electrostatically coated with PEEK powder, which later was melted at 380 ^{0}C, resulting in a homogeneous and highly adhesive coating. This layer, together with the aluminum substrate, were tested using a "calotest" abrasive wear (alumina 5 micrometer - water solution) and adhesive wear (AISI 52100 steel sphere) machine. In the case of the adhesive wear, the PEEK performance was far superior to that of aluminum. In the case of abrasive wear, aluminum substrate presented superior performance.