macroscopic stress, the following general equation can be written as [1]

$$\overline{}_{hkl} \quad \frac{1}{2} s_1^{hkl} [\overline{}_{11} \cos^2 \quad \overline{}_{12} \sin 2 \quad \overline{}_{22} \cos^2 \quad) \sin^2$$
$$\overline{}_{33} \cos^2 \quad (\overline{}_{13} \cos \quad \overline{}_{23} \sin \quad) \sin 2 \quad]$$
$$s_1^{hkl} (\overline{}_{11} \quad \overline{}_{22} \quad \overline{}_{33}) \qquad (3)$$

To determinate the particle size and the microstrain, the integral breadth was separated exactly as it is used in the single line Voigt function method [2]. Then the particle size and the microstrain may be obtained by:

$$D \quad \frac{K}{{}_c \cos} \,, \; e \quad \frac{{}_G}{4 \tan} \qquad (4, 5)$$

Where ${}_c$ and ${}_g$ are the Cauchy and Gaussin components of the integral breadth.

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Acknowledgements.
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P26

STUDY OF RESIDUAL STRESS OF A HIGH – ALLOY TOOL STEELS AFTER ELECTRO DISCHARGE MACHINING

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The objective of this contribution is investigation of residual stress state and distribution of microhardnes in surface layers of samples subjected to progressive unconventional technology electro discharge machining (EDM). Experimental samples were machined using graphite and electrolytic copper electrodes in common technological processes of finishing and stocking. Results of X - ray diffraction technique comply with the assumption that the layers machined by EDM exhibit isotropic biaxial residual stress state.

Introduction

The long-term experiences with applications of EDM and investigations of surface layers show that specific changes occur; their usual distribution is demonstrated in Fig. 1.

The intensity of removal of material during EDM is proportion to the energy of electrical discharge which is supplied by the generator of the machine tool. Except the energetic parameters (e. g. voltage, current, time of discharge) a large array of other factors determine the surface state and therefore its quality. Physical properties of the electrodes, which consist of the tool and the work-piece, belong among the most decisive parameters and hence the choice of polarity of discharge generator plays a crucial role. The aim of this contribution is to focus on the relation between the final state of the surface and the properties of electrodes, because the construction of generator and the recommendations of producers of tools vary.

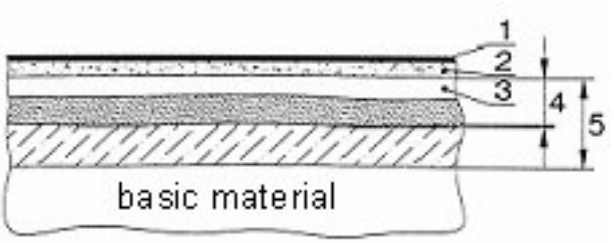

Fig. 1. Surface layer of steel affected by EDM [1].
1- microlayer of chemical compounds which originated during diffusion of dielectric, 2 – layer containing the material of tool electrode, 3 – the so called "white layer", strongly armorized resolidated melt, fine martensite-like structure, hardness 60 HRC, thickness from 0.04 mm to 0.3 mm depending on the energy of pulses, 4 – heat affected zone (hardened and tempered basic material of work-piece), 5 – plastic deformation zone induced by surges.

Samples under investigation

The experimental samples of dimensions 30 30 7 mm^3 were made from high-ally tool chrome steel ČSN 19436 (C 1.8 – 2.05 %, Mn 0.20 – 0.45 %, Si 0.20 – 0.45 %, P 0.03 %, S 0.035 %, Cr 11.0 – 12.5%, Ni 0.50 %). One half of the samples was left in basic state, the other was hardened according to the standard ČSN 41 436 [2] onto secondary hardness 58 - 60 HRC (heating in a vacuum furnace at austenitic temperature 1070 °C, hardening in oil bath and double tempering at 490 °C).

X-ray diffraction analysis and investigation of gradient of microhardnes were performed on the samples machined by EDM using generator with indirect polarity (tool +, work-piece -). This technology was carried out on the *WALTER Exeron S 204* with pulse generator with power of 9 kW and current of 180 A. Two modes of machining were used:

a) *finishing* was done by graphite and copper electrodes. The goal of finishing cycle was a surface of *Ra* 1,8 m.

b) *stocking was done by graphite and copper electrode. The machining system was set according to tables in order to achieve roughness Ra* 6,3 m. The working cycle is characterized by higher values of discharge time, open-circuit voltage and discharging current.

Analytical methods employed

An X-ray diffraction technique „sin^2 **"** [3] was realised by a Siemens -goniometer with CrK radiation. The diffraction line {220} Ni was analysed. Residual stresses were evaluated provided that the state of residual stresses was biaxial.

Measurement of microhardnes using method of chamfer cut [4] was performed with help of microhardnes testing machine *SHIMADZU HMV-2*. The conditions of microhardnes measurement according to Vickers HV 0.2 were: compressive force 1.961 N, time of load 12 s. Data obtained from microhardnes testing machine were evaluated by software LECO.

Conclusions

X-ray residual stress analysis

The biaxial isotropic state of residual macroscopic stress was identified in all cut surfaces machined by electro discharge machining with indirect polarity.

The results obtained by X-ray diffraction confirm the assumption of redistribution of energy in discharge canal. Compressive residuals stress on the cutting area machined by stocking using graphite end copper electrode contradicts the usually published results of trenching with direct polarity (tool – and working-piece +), where the residual stresses of EDM treated surfaces are tensile. This observed fact is probably caused by surplus of electrically positive elements in discharge canal and consequently different redistribution of energy between electrodes. The values of residual stresses correspond with different behaviour of microhardnes for indirect polarity.

Analysis of microhardnes behaviour

Unhardened samples machined by stocking, which means higher energetic values of discharge, exhibit growth of microhardnes in surface layers, which is observed by either electrode. In consequence of different electrical and thermal properties of electrodes, the graphite electrode leads to lower values of microhardnes and to lower depth of affected layer, approx. 30-50 m. The depth of affected layer by copper electrode is approx. 50-80 m.

As far as finishing is concerned, both electrodes are equal, because this technological mode does not influence microhardnes at all. The deviations of obtained values are in range of experimental error.

Hardened samples machined by stocking as a consequence of energy and heat removal in discharge canal exhibit nonstandard decrease of microhardnes in surface layers (graphite approx. 100 m, copper approx. 250 m), which is probably caused by structure changes and different energetic balance. This observed fact will be researched further and lateral metallographic specimens will be prepared.

In finishing mode, graphite electrode has no impact on microhardnes, while copper electrode leads to distinctively lower values of microhardnes in surface layer in thickness of 40 m. Structural changes, which arise due to machining using indirect polarity, are probably responsible for this behaviour (flow of electrically positive elements released from the tool electrode prevails).

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P27

CHARACTERISTICS OF DUERR IMAGING PLATE OPG

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Introduction

The goal of this study is to provide a relation between density of blackening and exposure for Duerr Imaging Plate OPG, which is used as a position sensitive detector in XRD laboratory of Department of Solid State Engineering. Comparison between photographic film and imaging plate from the point of view of linearity was performed. Backscattering Debye – Scherrer experiment with Ag standard was carried out in order to gain diffraction pattern on imaging plate. Dependences of background, absolute and relative peak height versus exposure time for two wavelengths of X-ray radiation were evaluated.

Density of blackening

Absorption of photon in the sensitive layer of silver bromide leads to formation of photographic latent image. The unexposed crystallites of silver bromide are removed in the fixing bath. Density of blackening D can be expressed as

$$D \quad \log I_0 / I \qquad (1)$$

where I_0 is the intensity of incident light and I is the intensity of light that passed through a developed and fixed photographic film. Density D may be expressed as a function of exposure time t and intensity of incident X-ray beam I_X. Hence the characteristics of film is given by $D = f(I_X*t)$, the maximum value of D where D is linear function of I_X*t varies from 0.5 to 2.5 [1].

Sensitive layer in imaging plate comprises of luminofore barium chromo-bromide, which is excited by incident photon into a semi-stable state. By an illumination with He-Ne laser the process of photostimulated luminescence is triggered and the image in form of 16-bit grayscale pattern is released. Now I_0 in eq. (1.1) is the maximum value on grayscale $2^{16} = 65536$ and I is the information in the chosen pixel. It can be still assumed that D is function of exposure time t and intensity of incident X-ray beam I_X.

Experiment

The backscattering diffraction experiment was done using CuK and CrK radiation, the exposure times varied from 1 to 20 minutes for copper anode and from 1 to 55 minutes for chrome anode. The plate holder was rotated at 1 rpm in order to avoid effects of coarse grain of the standard Ag. The incident beam impinged the sample in direction normal to its surface. The 16-bit diffraction pattern on the imaging plate was obtained by scanning on VistaScan by Duerr. Lucia 5.10 image analysis system was used to gain intensity profile, which was transformed into density profile employing eq. (1.1).

Evaluation of profiles

In Fig. 1 density profile is depicted, dependences of following parameters on exposure time were investigated: absolute and relative diffraction peak height, integration intensity, FWHM and level of background. The peaks were approximated by Gaussian function $D = a + b * exp(-0,5((p-c)/d)^2)$, where p is position in pixels, and background by two linear functions $D = K_1p + Q_1$, $D = K_2p + Q_2$. Value D_{lin} as a maximum density, where linear relation between D and t is observed, was figured out for both wavelengths.

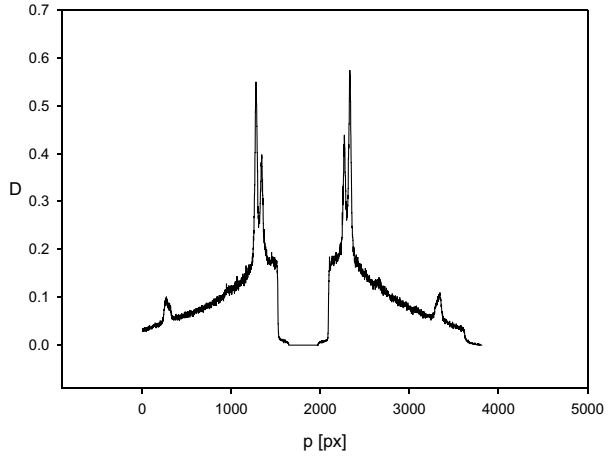

Fig. 1. Density profile of imaging plate exposed for 13 min by CuK .

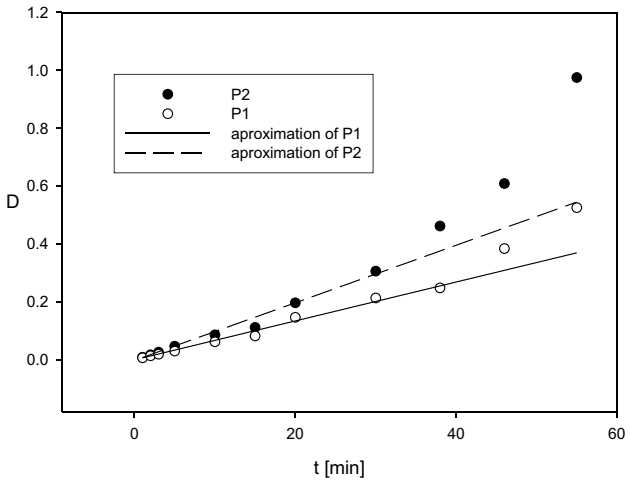

Fig. 2. Absolute peak heights for CrK .

Conclusions

Following statements can be derived from computed characteristics of imaging plate:

(i) Value $\boldsymbol{D_{lin} = 0.3}$ was established. That corresponds to the intensity range (34000, I_0) in 16-bit image.

(ii) Photographic films exhibit effect of solarization for $D > \boldsymbol{D_{lin}}$, when the level of blackening declines by big exposures. Whereas imaging plate display higher values of density for $D > \boldsymbol{D_{lin}}$ than would correspond to linear evolution as can be seen in Fig. 2.

(iii) If the absolute peak height is less than $\boldsymbol{D_{lin}}$, the plots I_{int} *(t), b (t)* are linear. For $D > \boldsymbol{D_{lin}}$ deviations from linearity occur.

(iv) No obvious relation between FWHM and exposure was found.

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P28

ON THE TRANSITIONS OF THE 2H MARTENSITE SINGLE VARIANT OF CuAlMn ALLOY

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The method of successive compression loads to different faces of prism shaped shape memory alloy (SMA) single crystal specimens has been presented as an efficient approach to investigate twinning deformation processes in martensitic phases [1]. This approach (developed originally on CuAlNi single crystals) is applied in the present work and is concerned with the investigation of the twinning processes in the 2H martensite phase in CuAlMn SMAs.

Two single crystalline CuAlMn alloys of various chemical compositions (e/a ratio) in a form of prismatic samples were subjected to compression experiments [2]. Twinning deformation processes in the martensite were investigated by the i) compression stress-strain responses recorded for multiple load axis orientations, ii) measurements of shape changes due to the reorientation of martensite single variants, and iii) the three surface trace analysis method for the characterization of twining planes. The experimental results were discussed and compared with theoretical calculations of the type of twinning, a value of twinning stresses, its orientation dependence etc. Finally, the results of stress-strain behavior of this alloy were compared with behavior of CuAlNi shape memory alloy and is shown the advantage of the shape changes measurements according to the martensite variant determination where the variant existing in the sample can be clearly determined in comparison with other methods (Back Laue reflection method).

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P29

REORIENTATION PROCESSES IN CuAlNi SINGLE CRYSTALS STUDIED BY NEUTRON DIFFRACTION TECHNIQUE

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The microstructure of martensites created by thermally induced martensitic transformation from austenite single crystal consists of multiple habit plane variants arranged in a self-accommodated manner - i.e. the martensite sample containing large amount of internal interfaces is not a martensite single crystal. However, when such a crystal is deformed in martensite state (e.g. by successive compression deformations applied to different faces of a prism shaped sample [1]), the internal interfaces can be driven out of the sample and a true single crystal of the martensite phase may thus be prepared. Before such a relatively large martensite single-crystal is further utilized for e.g. measurement of martensite elastic constants by ultrasonic methods [1], its quality (whether the sample is really free of twin interfaces) needs to be checked.

One can inspect the polished faces of the martensite prisms for traces of interfaces, compare the measured shape of the martensite prism with a theoretical prediction [1], or take the linear compression stress-strain response as indirect evidences for the singlecrystalinity of the sample. Neutron diffraction yielding bulk structural information from a sample fully immersed in the neutron beam, on the other hand, may be used to provide direct evidence.

In this work, we describe a neutron single crystal diffraction approach recently developed for inspecting the quality of 2H martensite single crystals of CuAlNi alloy prepared from originally cuboid shape austenite single crystal samples. The experiments were carried out on a single crystal diffractometer at NPI Rez using monochromatic neutron radiation (wavelength 1.44 Å) equipped with a ^{3}He counter, 2D imaging plate detector and miniature screw driven deformation rig placed in a 3-axis goniometer. Neutron diffraction results show that it is in fact quite difficult to prepare sufficiently good martensite single crystals, since the martensite twinning processes leading ultimately to single martensitic variant are often not completed, even if the indirect methods seem to suggest that the sample already exists in the desired martensite single crystal form.

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P30

NEUTRON DIFFRACTION ANALYSIS OF RETAINED AUSTENITE IN Mn-Si TRIP STEEL DURING PLASTIC DEFORMATION

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TRIP (Transformation Induced Plasticity)-aided multiphase steels are promising structural materials with well balanced properties, combining high yield strength and excellent formability. Structural materials with such beneficial mechanical properties are required particularly by the automotive industry for the manufacture of light but strong components which also provide good energy absorption during dynamic loading. The combination of high strength and ductility is attributed to the transformation-induced plasticity effect resulting from the strain-induced martensitic transformation of the retained austenite in the ferrite-bainite microstructure.

P31

NEUTRON DIFFRACTION STUDIES OF AUSTENITE-TO-FERRITE TRANSFORMATIONS IN LOW-ALLOY STEELS

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Production of construction materials with well balanced strength and toughness is a many years challenge task for material designers. Taking into account also economic aspects, one of the promising ways is a production of the multiphase low-alloyed steel with favorable phase composition and fine grain microstructure yielded by a special concept of thermo-mechanical processing. In the present work, the austenite-to-ferrite phase transformation in low alloy steels was studied by neutron diffraction methods. This method was used *in situ* during thermo-mechanically controlled processing /TMCP/ to study transformation kinetics and behavior of phase lattice strains. Moreover, the neutron diffraction is also used for monitoring of texture evolution during TMCP. Two types of steels containing different amount of Nb addition were examined. The influence of production parameters such as deformation degree and Nb addition on the steel microstructure and its mechanical properties is reported in particular.

Neutron diffraction was used to examine the TRIP effect in different thermomechanically treated steel samples. In order to evaluate the effect of the applied thermomechanical processing parameters on the multiphase structure development and subsequently on retained austenite stability, samples were studied in-situ during tensile loading at room temperature using the ENGIN-X diffractometer at the ISIS neutron source. Neutron diffraction has been found to be a convenient method for the characterization of transformation kinetics of strain-induced martensitic transformation in TRIP steels during mechanical loading. The evolution of transformation stresses with applied external load was determined in the retained austenite and ferrite phases. The relationship between yield strength of TRIP steel and volume fraction of retained austenite was found.

P32

MICROSTRUCTURE DEVELOPMENT IN Cr-Al-Si-N NANOCOMPOSITES

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During the last years, the importance of thin films nanocomposites for technological applications increased rapidly. Nanocomposite thin films are considered as a suitable material for protection of cutting tools used in high temperature applications. The required properties of such thin films are, in particular, high hardness and good chemical stability at temperatures exceeding 1000 °C.

In this contribution, we describe the microstructure development in the Cr-Al-Si-N nanocomposites with different [Cr]/([Al]+[Si]) ratio.

The Cr-Al-Si-N coatings were deposited using cathodic arc evaporation in nitrogen atmosphere with the working pressure of 1.3 Pa using two laterally rotating arc cathodes (-80 from PLATIT) [35]. One cathode was made of chromium, the second one from aluminum containing 11 at % Si. The ion current on the Cr cathode was 80 A, on the Al-Si cathode 120 A. The bias voltage was -75 V. Polished plates of cemented carbide were used as substrates. The base pressure was 5 10^{-3} Pa; the deposition temperature was approximately 450 °C. In contrast to commercial coatings, the samples were not rotated during the deposition process, which offers the following advantages for microstructure studies. The expected preferred orientation of crystallites in $Cr_{1-x}Al_xN$ is not superimposed by the sample rotation. A series of coatings with different chemical compositions can be obtained in one deposition process, as the chemical composition depends on the distance from the respective cathode.

The samples were investigated using electron probe microanalysis with wavelength-dispersive spectroscopy, X-ray diffraction and high-resolution transmission electron microscopy. The following topics are discussed in particular: phase stability of the Cr-Al-Si-N system, crystallite size, preferred orientation and crystallographic coherence of crystallites, the crystal anisotropy of the X-ray elastic

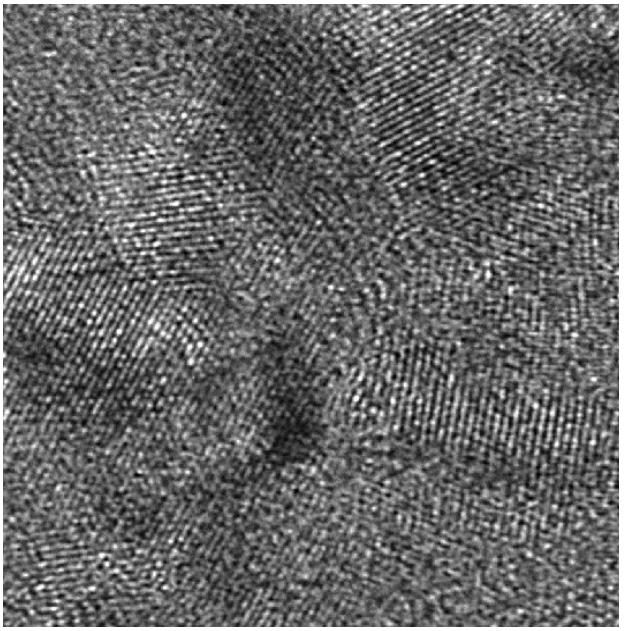

Fig 1. HRTEM micrograph of the sample $Cr_{0.40}Al_{0.52}Si_{0.08}N$ showing nanocrystallite particles.

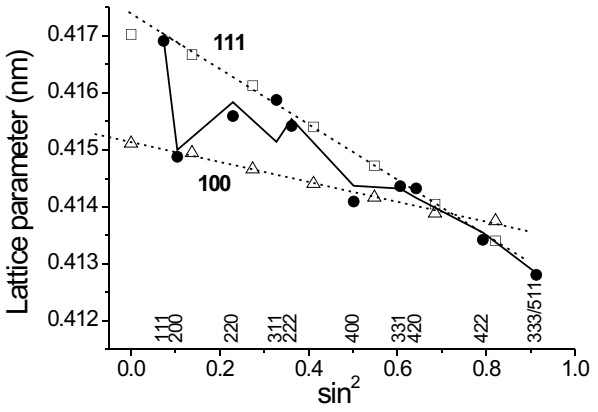

Fig 2. Sin^2 -plot for the lattice parameter measured in the fcc phase of the sample Cr0.91Al0.08Si0.01N showing large crystal anisotropy of the lattice deformation. Open symbols show the lattice parameters obtained from the interplanar spacings of the lattice planes (111) and (200), filled symbols the lattice parameters measured using GAXRD on different lattice planes. For the GAXRD method, the diffraction indices are given at the bottom of the sin、 -plot.

constants and the interplay between the microstructure and the hardness.

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P33

MODIFICATION OF UHMWPE CRYSTALLINE STRUCTURE BY MEANS OF e-BEAM IRRADIATION AND THERMAL TREATMENT

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Ultra-high molecular weight polyethylene (UHMWPE) is used as a key component of artificial human joints, such as hip and knee, due to its balanced mechanical and friction properties. Nevertheless, the wear of UHMWPE, i.e. the release of microscopic particles from the polymer surface, seems to be the main reason why total joint replacements (TJR) fail. The wear particles move from the joint space to the surroundings of TJR, where they cause inflammatory reactions and osteolysis. In recent years it has been demonstrated that UHMWPE wear resistance can be increased by means radiation-induced crosslinking.

In this study, bulk UHMWPE was irradiated with accelerated electrons (doses from 0 to 100 kGy, dose rates > 25 kGy/h) to crosslink the polymer and thermally treated above the melting point (T_m = 140 °C) to eliminate residual macroradicals and to limit oxidative degradation. Level of crosslinking was checked by solubility experiments and extent of oxidation was investigated by spectroscopic methods (IR, EPR). Irradiation and thermal treatment result in considerable changes in both molecular and supermolecular structure of UHMWPE, which influences not only its wear resistance, but also other mechanical properties. We followed the structural changes by small- and wide-angle X-ray scattering (SAXS and WAXS). Supplementary pieces of information were obtained also by differential scanning calorimetry (DSC) and scanning electron microscopy (SEM). We proposed model of supermolecular structure changes after irradiation and/or thermal treatment. The model is based on quite simple assumption that UHMWPE is composed of three phases: crystalline, amorphous and crosslinked.

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