Materials Science Forum Vols. 571-572 (2008) pp 101-106
online at http://www.scientific.net

Online available since 2008/Mar/07

Temperature Dependence of Residual Stress Gradients in Shot-Peened Steel Coated with CrN

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Keywords: XRD, high temperature, residual stress gradient

Abstract. A temperature behaviour of residual stresses in shot-peened steel coated with 3μm CrN is characterized using in-situ energy dispersive synchrotron X-ray diffraction performed in the temperature range of 25-800°C. The samples are thermally cycled and the development of volume-averaged residual stresses in the coating and residual stress depth gradients in the steel is characterized. The results reveal complex changes of stresses in CrN and in the substrate. The annealing results in the removal of stress gradients in the steel which starts at the temperature of about 600°C. After cooling down, there are no stresses detected in the steel. The temperature dependence of stresses in CrN is very complex and indicates the presence of phenomena like an annealing of intrinsic stresses about the deposition temperature of 350°C, a formation and a closing of micro-cracks in the tensile region and finally a stress relaxation of approximately 500 MPa after the cooling down. The presented approach allows a complex characterization of thermo-mechanical processes in coating-substrate composites and opens the possibility to understand phenomena related to the thermal fatigue of coated tools.

Introduction

Hard coatings based on CrN are routinely used to protect bulk materials from abrasion and corrosion [1,2]. The successful application of those coatings demands an optimisation of thermal fatigue resistance in order avoid cracking or rupture of the coating [3, 4]. The optimization resides primarily in the engineering of coating nano-structure, residual stress state, interface properties and substrate pre-treatment [5-8].

Residual stresses in coatings and in the surface region of substrates influence significantly mechanical properties and lifetime of coated working tools [9]. Hard and super hard coatings are expected to exhibit very high compressive stresses in the surface region what increases wear resistance whereby relatively small compressive stresses at the interface region should help to avoid coating rupture. It is believed that high-compressive stresses in the substrate surface region contribute also to the improved fatigue resistance of working tools. Surface stresses in tool steels are usually produced by shot peening what results in an increased resistance to crack initiation and propagation [10-12].

For the production of coated working tools with a good thermal fatigue resistance, it is important to select coating-substrate systems with relatively stable structural properties at high temperature. A common way to look at the temperature-induced changes in the structure and in residual stress state is to perform *in-situ* X-ray diffraction (XRD) experiments. Using this approach, it is possible to monitor structural changes at the conditions similar to working ones and assess phenomena contributing to the degradation effects in hard coatings and in substrates.

For the characterization of residual stress gradients in the near surface region of polycrystalline materials, an application of energy dispersive X-ray diffraction (ED-XRD) was recently proposed [13,14]. The main advantage of that technique resides in the fact that individual diffraction lines *hkl* are recorded at different energies *E*(*hkl*). In this way, each reflection and the corresponding $\sin^2\psi$ plot can be correlated with a specific penetration depth $\tau(hkl)$. Since the synchrotron ED-XRD analysis of polycrystalline materials can be performed relatively fast, the technique is ideal for time-resolved studies of residual stress fields [13,14].

In this work, in-situ synchrotron ED-XRD was used to characterize residual stresses in a system CrN coating-shot peened steel. The structures were thermally cycled and volume-averaged residual stresses in the coating and residual stress depth gradients in the steel were evaluated as a function of temperature. The aim was to understand at which temperature an irreversible relaxation of stresses in the coating and in the substrate occurs.

Experimental

Specimen Preparation. As a substrate for the specimen preparation, sheets of shot-peened ferritic steel B316 with the thickness of 1 mm obtained from a commercial partner Böhler Ybbstahl (Böhlerwerk, Austria) were used. The substrates were coated with a 3µm CrN coating in an Ar+N_2 atmosphere at 350°C using a bias voltage of -80 V.

Laboratory Measurements. Residual stress depth profiles in the shot-peened ferritic steel were characterized using laboratory XSTRESS 3000 diffractometer. The measurements were performed using Cr-K_α radiation and a collimator of 3 mm. During the measurement procedure, layers of the plastically deformed material in the subsurface region were removed stepwise with a step of about 20 µm by chemical etching. At each step, the in-plane residual stress was determined by the diffraction.

Synchrotron Measurements. In-situ residual stress characterization of stresses was performed at *Energy Dispersive DIffraction* (EDDI) beamline of BESSY in Berlin, Germany. Samples of steel coated with CrN were thermally cycled in the range of 25-800°C using an Anton Paar heating chamber DHS 900 at constant heating and cooling rates of 0.3°C per second [15]. During the thermal treatment, structural properties of the samples were characterized using ED-XRD. The measurements were carried out with a white beam of the energy range 20-100 keV. For the data acquisition, a N_2 cooled LEGe detector system from Canberry with a resolution of 160 eV at 10 keV and 420 eV at 100 keV was used. The acquisition was performed at constant 2θ angle of 14° in symmetric $\theta/2\theta$ configuration with a counting time of 10 seconds per one recorded spectrum. Three reflections of CrN (111,200,220) and six reflections of ferritic steel (110,200,211,220,310,222) were detected simultaneously in one spectrum. The correlation between the lattice spacing $d_\psi^T(hkl)$ (measured at the temperature T and the sample tilt angle ψ) and the corresponding diffraction line $E(hkl)$ is given by

$$d_\psi^T(hkl) = \frac{hc}{2\sin\theta}\frac{1}{E(hkl)} = const\frac{1}{E(hkl)}\,. \qquad (1)$$

[14], whereby h is the Planck's constant and c is the velocity of light. The penetration depth τ for a reflection *hkl* can be calculated according

$$\tau(hkl,\theta) = \frac{\sin\theta}{2\mu E(hkl,\theta)}\cos\psi\,\cdot \qquad (2)$$

[14], where μ is the absorption factor of the material, E is the energy of the reflection *hkl* calculated using Eq. 1 and ψ is the angle between the sample normal and the diffraction vector. The measurements were performed using a $\Delta\sin^2\psi$ step of 0.1 [15]. From the measured lattice spacing $d_\psi^T(hkl)$, temperature-dependent in-plane isotropic residual stresses in the CrN coating and in the substrate $\sigma^T(\tau)$ were evaluated according

$$d_\psi^T(hkl) = d_0(hkl)\left(1+\sigma^T(\tau[hkl])\ [2s_1^T(hkl)+\frac{1}{2}s_2^T(hkl)\sin^2\psi]\right). \tag{3}$$

whereby $s_1^T(hkl)$ and $\frac{1}{2}s_2^T(hkl)$ are the X-ray elastic constants (XECs) of the material. It was supposed that the stresses are in-plane isotropic with $\sigma^T = \sigma_{11}^T = \sigma_{22}^T$. XECs for CrN and ferritic iron were calculated from the single-crystal elastic constants (SECs) assuming Hill model and using the software ElastiX [16].

ED-XRD characterization of CrN coated steel was applied to six samples. The results from all samples were comparable. Here below, representative results are presented.

Results and Discussion

Representative residual stress depth profile in the B316 shot-peened steel determined in laboratory conditions is presented in Fig. 1. The results document a typical dependence of stresses on depth in the shot-peened material.

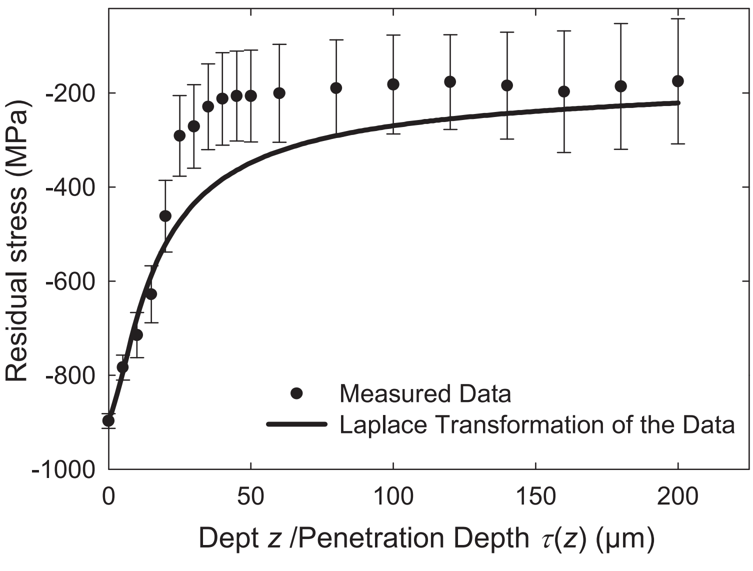

Fig. 1. Depth dependence of residual stresses $\sigma(z)$ in the shot peened steel of B316 obtained using laboratory procedure. The circles represent measurement points. The solid line indicates Laplace transformation $\sigma(\tau)$ of the measured data.

Relatively high compressive stresses decrease exponentially and approach the stress free level (Fig.1). It can be expected that, at the larger depths, the stresses become even tensile. For a comparison, a Laplace transformation $\sigma(\tau)$ of the measured profile $\sigma(z)$ is plotted. The calculated dependence was obtained by a numerical integration of the data $\sigma(z)$ from Fig. 1 according

$$\sigma(\tau) = \frac{1}{\tau}\int_0^\infty \sigma(z)e^{-z/\tau}\mathrm{d}z\,. \tag{4}$$

An obvious similarity between $\sigma(z)$ and $\sigma(\tau)$ (Fig. 1) can be explained by the specific exponential nature of the measured stress dependence $\sigma(z)$.

Since the ED-XRD procedure can be used to obtain residual stress as a function of the penetration depth $\sigma(\tau[hkl])$ in a non-destructive way, the calculated profile $\sigma(\tau)$ in Fig. 1 represents an important indicator of the procedure reliability. Moreover, changes in the experimental $\sigma^T(\tau[hkl])$ can be effectively correlated with the changes in the dependence $\sigma^T(z)$. In Fig. 2, a temperature dependence of residual stresses on the penetration depth $\sigma^T(\tau[hkl])$ in a CrN coated shot-peened steel measured during one temperature cycles is shown.

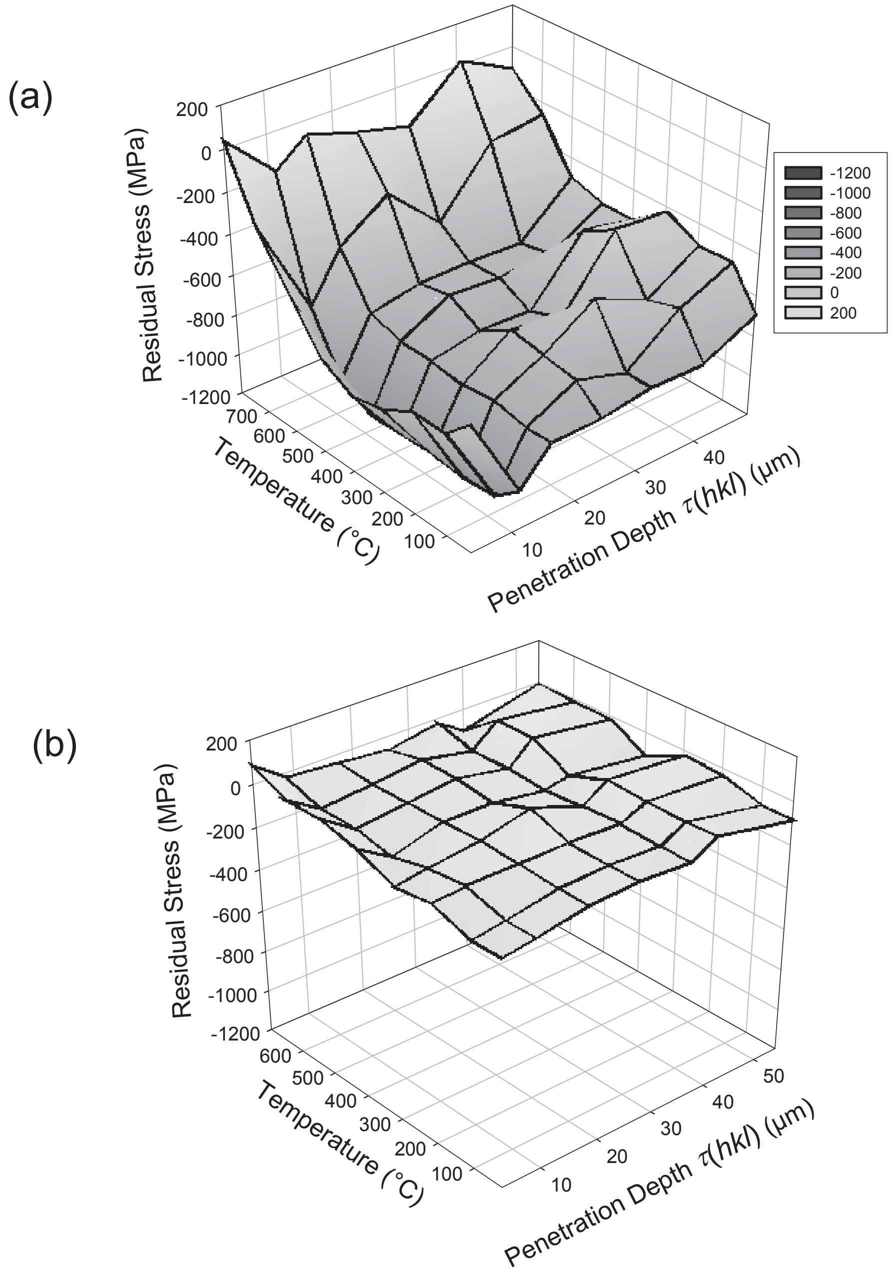

Fig. 2. A temperature dependence of residual stresses σ^T in a CrN coated shot-peened ferritic steel as a function penetration depth $\tau(hkl)$ during heating up (a) and cooling down (b).

Fig. 2a indicates that the temperature increase from 25 upwards results in the expected decrease of the residual stresses in the steel. The stress relaxation becomes very intensive especially at the temperatures above 600°C. At 800°C, there is practically no residual stress present in the substrate.

In contrast to the $\sigma(\tau)$ dependence from Fig. 1, the maximal stress in $\sigma^T(\tau[hkl])$ dependence is not observed at the surface but at about 15µm of penetration depth. A similarity between $\sigma^T(\tau[hkl])$ at 25 °C (Fig. 2a) and $\sigma(\tau)$ (Fig. 1) suggests that the annealing of the shot-peened substrate results in the stepwise remove of the exponential stress dependence. The temperature change during cooling down (Fig. 2b) does not induce any significant changes and the stress free state (± 50 MPa) is preserved. Also additional annealing of the sample (2nd cycle) did not induce any changes in stress. From the technical point of view, the temperature of 600°C seems to be critical for the application of tools based on the shot-peened steel B316. Above the critical temperature, the annealing can negatively influence the mechanical performance of those tools.

Simultaneously with the characterization of the stress profiles $\sigma^T(\tau[hkl])$ in the substrate, also temperature dependence of the macroscopic residual stress in CrN coating (Fig. 3) was evaluated (Eq. 3) from the ED-XRD spectra using the strain data from CrN 111, 200 and 220 reflections. Since the absorption of the synchrotron beam in the 3µm CrN is negligible, only volume-averaged stress values $\sigma^T(\tau = 0)$ were refined using Eq. 3.

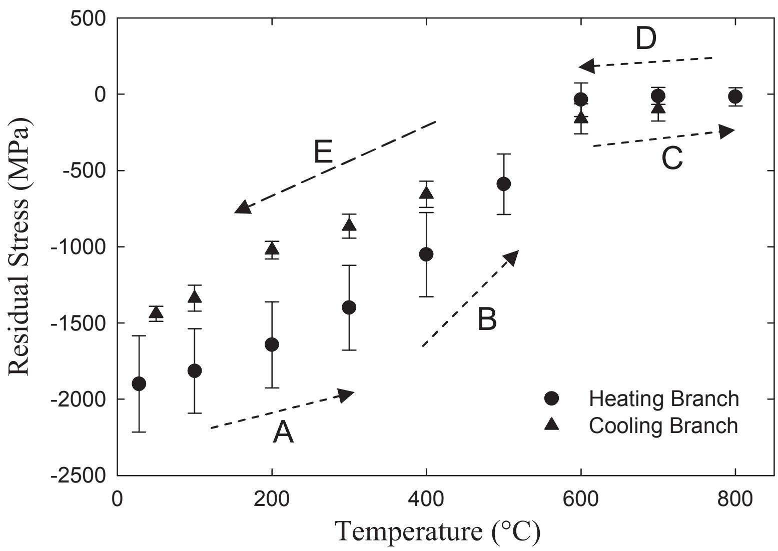

Fig. 3. A temperature dependence of residual stress in CrN coating on shot-peened steel during one temperature cycle. Behaviour related to A-E segments is discussed in the text.

In the temperature dependence of residual stresses in CrN on steel, one can identify 5 segments. In the region denoted as A, a compressive stress in CrN decreases thermo-elastically since thermal expansion coefficient (TEC) of CrN is smaller than that of iron [12]. In region B above the deposition temperature of 350°, an annealing of intrinsic stresses caused by the presence of point defects starts. In the region C, the coating stress switch to tensile and one can expect a formation of micro-cracks in the ceramic-like coating. When the cooling down starts, at first the cracks in the coating are being closed (region D). After this effect is completed, a compressive stress is formed in the coating as a result of TEC mismatch. After the cooling down, one can observe a stress relaxation of about 500 MPa what can be attributed to the phenomena observed in the segment B. During a second temperature cycle, there was no stress relaxation observed (region B). In other words, there was no stress-temperature hysteresis detected during second thermal cycle.

Conclusions

ED-XRD was used to characterize temperature dependence of stresses in the system CrN coating - shot-peened steel. The structures were thermally cycled and the stresses in CrN as well as stress gradients in the substrate were evaluated as a function of the temperature. The results indicate a complex behaviour of stresses in the coating with a typical stress-temperature hysteresis. In the case of substrate, the annealing results in the annealing of the residual stress gradients especially above 600 °C. During the cooling down, the stresses in CrN are partly recovered whereby the substrate remains practically stress free. The presented approach demonstrates for the first time a possibility to characterize simultaneously complex residual stress states in coating and in the underlying substrate during one temperature cycle and opens the possibility to understand phenomena related to the thermal fatigue of coated tools.

Acknowledgment

The authors are grateful to the group of Prof. Christoph Genzel from EDDI beamline for the support during ED-XRD measurements. This work was supported by the Austrian NANO Initiative via a grant from the Austrian Science Fund FWF within the project "StressDesign - Development of Fundamentals for Residual Stress Design in Coated Surfaces." Additionally, this work was supported by the European Community - Research Infrastructure Action under the FP6 "Structuring the European Research Area" Programme (through the Integrated Infrastructure Initiative" Integrating Activity on Synchrotron and Free Electron Laser Science - Contract R II 3-CT-2004-506008).

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