EVIDENCE AND ANALYSIS OF THERMAL STATIC STRAIN AGING IN THE DEFORMED SURFACE ZONE OF FINISH-MACHINED HARDENED STEEL

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ABSTRACT

After heat treating, finish machining of the hardened steel represents the last manufacturing step of machine elements. The practically most important operation of grinding is applied to achieve edge zone compressive residual stresses, best surface quality and dimensional accuracy. Metal removal involves high plastic deformation work. Glide and intersection processes raise the density and produce lower energy substructures of dislocations. The temperature and time behavior of post-machining thermal treatment is analyzed on ground and honed martensitic SAE 52100 rolling bearing steel. Microstructure stabilization is reflected in a large XRD peak width decrease on the surface. The kinetics are modeled by rate-controlling carbide dissolution as the carbon source for Cottrell-type segregation at dislocations. This thermal static strain aging is verified by the formation of a slight white etching surface layer. The model is extended to also consider superimposed thermal dislocation recovery. Both effects are separable. In rolling contact fatigue tests under mixed friction conditions, air reheating below the tempering temperature, which avoids hardness loss, leads to a significant lifetime increase. The effect also occurs after cold working.

INTRODUCTION

As the final production step of engineering parts, hard machining completes mechanical shaping and provides sufficient roughness. In the present paper, grinding and honing, i.e. fine grinding, is applied to bearing ring raceways. In an adequately cooled process [1], strong plastic deformation leads to the formation of crack inhibiting compressive residual stresses in the edge zone. Characteristic surface values after grinding and honing of hardened steels equal around –600 to –500 MPa. Glide processes result in the development of low-energy dislocation substructures. These spatial configurations, like dipoles and multipoles, stabilize the microstructure. Despite the increased dislocation density, therefore, for hardened steels the X-ray diffraction (XRD) peak width is reduced by surface finishing and cold working in the plastically deformed edge zone.

Grinding represents a well-established, widely used abrasive process [2]. It is mainly applied as a hard finishing operation with only little material removal. The thickness of the mechanically influenced near-surface zone is no more than 10 µm. New developments aim at including extended shaping work steps [3], comparable, for instance, with hard turning. Other process optimization efforts tend to exploit the induced microstructure stabilization by an added value operation of reheating the ground steel components [4]. In the present paper, this post-machining thermal treatment (PMTT) is analyzed experimentally and described by simulations over an enlarged temperature range based on an extended metal physics model.
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STATE OF KNOWLEDGE AND MOTIVATION OF THE PRESENT WORK

In a recent paper [5], one of the authors has published results on the PMTT effect on ground and honed roller bearing inner rings of martensitically and bainitically hardened steel SAE 52100. To introduce an economical process and avoid hardness loss, this study focuses on short air reheating below the tempering or transformation temperature. The small reduction of the surface compressive residual stresses by around 50 MPa is negligible for the application. The retained austenite content remains unchanged. However, the significant decrease of the (211) ferrite XRD peak width, measured with Cr-Kα radiation, on the surface with reheating temperature and time, detected for each heat treatment, points to substantial microstructure stabilization in the outermost material zone: the large reduction by up to 0.5° clearly exceeds the typical lessening of 0.2° by this finishing process itself. The changes of the residual stresses and peak width by the thermal post-treatment are essentially limited to the surface [5], which agrees with the small mechanically influenced zone by grinding and honing. The formation of a slight white etching layer by reheating the finished parts supports the microstructural reinforcement. The XRD peak width change on the surface describes the intensity of post-grinding thermal treatments. A microscopic model has been established, from which a kinetics equation for the simulation of this characteristic parameter is deduced. To verify the metal physics approach and the derived exponential decrease of the surface peak width with time, an extensive experimental study of isothermal reheating of ground and honed martensitic SAE 52100 steel is carried out in the present work. Also, rolling contact fatigue (RCF) tests under high surface loading are performed to estimate the effect of the post-grinding thermal treatment on the component lifetime.

SAMPLE PREPARATION AND EXPERIMENTAL METHODS

After standard austenitizing and quenching of SAE 52100 steel (German grade: 100Cr6), two martensite microstructures, LT and HT, are prepared that correspond to low and higher tempering for 2 h at 165 °C and 240 °C with typical hardness of 64 and 60 HRC, respectively. Actually, the HT material is produced by an equivalent short-cycle process at 300 °C for 15 min. Segments of inner and outer rings of cylindrical roller bearings with ground and honed raceways are reheated. XRD measurements of the full width at half maximum, FWHM, of the (211) ferrite reflection are performed applying Cr-Kα radiation with mean penetration depth of around 5 µm [6, 7]. Changes due to reheating are determined by comparing to the same sample in the initial as-finished state as reference. Mean value and standard deviation are derived from eight individual measurements. White etching areas are analyzed by light microscopy of metallographic cross-sections. Temper carbides are characterized by transmission electron microscopy (TEM) on carbon extraction replicas. Rolling contact fatigue tests are carried out at a 4-ball rig under a mean Hertzian pressure of 5 GPa at 1500 rpm rotational speed of the driven top part on surface finished specimens of 12.7 mm diameter made of LT material that are respectively reheated at 150 °C for 30 min and, for reference, not thermally post-treated. Each Weibull curve is derived from five repetitions. Mixed friction running conditions under poor oil lubrication result in a surface sensitive overrolling test.

RESULTS FOR LOW TEMPERED (LT) MARTENSITIC SAE 52100 STEEL

Due to process variations, the as-finished value of the surface peak width, FWHM_{t=0}, of the low tempered material LT ranges from 6.6 to 7.1°. These deviations influence the change with reheating time t at a certain temperature: ΔFWHM_{t=0}(t)=FWHM_{t=0}(t)−FWHM_{t=0}(t=0). Figure 1a
provides an illustration for largely different initial peak widths of 6.67 and 7.08° at the reheating temperature $T=160$ °C. Here, $z$ denotes the depth. The model equation used for data fitting and evaluation is derived in the next but one paragraph. The effect of the reheating temperature on the surface peak width change with time can best be demonstrated on uniform initial states, i.e. equal FWHM$_z(0)$ values. This is exemplarily done in Fig. 1b at 140 and 180 °C. The kinetics of the thermal surface XRD peak width reduction are measured in steps of 10 °C for moderate reheating temperatures from 140 to 180 °C: the detailed evaluation presented later in the text also distinguishes between two groups of similar low and high initial values. Within the considered reheating time of 2 h, all curves tend towards a certain saturation level that, with increasing temperature, is reached faster and reveals a higher absolute value, the maximum of which exceeds 0.4°.

![Fig. 1a, 1b. Isothermal surface FWHM change with time $t$ for several reheating temperatures $T$ of material LT: a)/b) Different/equal initial FWHM$_z(0)$ and equal/different $T$ values.](image)

Reheating produces a slight white etching layer in the surface zone plastically most deformed by the finishing process. Figure 2a presents an angled cross-section: the actual depth of the layer is around 1 µm. The same effect but with larger plastified region occurs on a hardness indentation; cf. Fig. 2b. Both, size and intensity of the white etching area increase with reheating temperature.

![Fig. 2a, 2b. Etched cross-sections of reheated material LT: a) PMTT at 180 °C for 2 h after the grinding and honing process. b) Heating of a Rockwell C indent at 160 °C for 2 h.](image)

**RESULTS FOR HIGHER TEMPERED (HT) MARTENSITIC SAE 52100 STEEL**

The reduction of the surface XRD peak width on the ground and honed samples of material HT is investigated for isothermal reheating below and above the tempering temperature of 240 °C. Figure 3a illustrates the time development in the range spanning 180 to 280 °C and the typical scattering of the data points. The initial peak widths of the as-finished samples are in the range of 5.5°. In Fig. 3b, the final values after reheating for 2 h reveal the influence of the temperature.
Fig. 3a, 3b. Change of the line width on finished surfaces of HT samples by isothermal reheating:

a) Time curves.

b) Reduction after 2 h as a function of temperature with trend line.

The measured kinetics curves are compiled in Figs. 4a and 4b. For a maximum reheating time of 2 h, the development of apparent saturation levels, which are reached faster and correspond to an increasing surface peak width reduction from 0.2 to more than 0.4° with rising temperature, again is obvious (cf. Fig. 3b). The course of the 280 °C curve following the initial steep drop above 30 min, however, suggests a subsequent slower further decrease, as indicated by an arrow in Fig. 4b.

Fig. 4a, 4b. Surface XRD peak width change of ground and honed HT specimens with reheating time up to 2 h: a) Below tempering temperature. b) Above tempering temperature.

THERMAL STATIC STRAIN AGING AND MODELING OF THE PMTT EFFECT

Low temperature oxidation during reheating in air forms a brownish oxide layer on the steel that may improve the wear resistance to a slight extent. However, it is not responsible for the large reduction of the XRD line width on the ground and honed surfaces since similar tempering colors also arise at the pre-electropolished areas in the depth of the material where no such FWHM change occurs [5]. For an Fe-0.81 wt.% C alloy oxidized in 133 hPa of dry oxygen, an oxide film thickness between 10 and 40 nm is reported for temperatures from 200 to 300 °C after 2 h [8]. In the two-stage logarithmic growth, at first only Fe₃O₄ is formed, followed by α-Fe₂O₃.
There are three contributions to the total broadening of the XRD peak: the instrumental part is not influenced by reheating. In hardened steels, the size of the crystallites, i.e. the coherently scattering regions, in the martensitic phase equals around 100 to 200 nm. The peak width is thus not directly correlated with the austenite grain size of few µm. Lattice distortion provides the dominating contribution to the high line broadenings in hardened steels, which is essentially not influenced by carbide precipitations. The large peak width reduction on ground and honed surfaces by reheating reflects microstructural stabilization in the edge zone of the material that is mechanically highly loaded in most applications. Since it occurs below the tempering or transformation temperature without hardness or retained austenite change and only on the finished surfaces, it is not caused by lattice relaxation due to retempering with further carbide precipitation. Also, static recovery, which results in a decreased XRD peak width because of the reduction of the dislocation density by network coarsening, does not explain the PMTT effect that depends on the heat treatment and appears rapidly at about 23 % of the absolute melting temperature of iron where no dislocation rearrangement occurs. Moreover, the formation of a slight white etching surface layer cannot be understood by either of these two approaches.

A reduced fraction of temper carbides causes the appearance of white (i.e. non-) etching microstructures in metallographic cross-sections of hardened steels. Dislocation lines, densely formed uncovered in low-energy configurations by hard surface finishing, strongly trap carbon atoms that mainly stem from the dissolution of temper carbides. In the plastified edge zone, these precipitations are, moreover, likely deformed or cut and thus destabilized by increased surface curvature. Figure 5 shows a TEM image of the typical temper carbide distribution in the core of LT material: with diameters from 10 to 50 nm, these particles are large enough to partly dissolve without loss of strength (Orowan mechanism valid) at moderate reheating temperatures and times. The connection with the temper carbides also explains the influence of the different heat treatments. Thermal static strain aging (TSSA) by Cottrell-type carbon segregation agrees with results for warm shot peening [9, 10]. Dislocations strongly pinned in their favorable arrangement to clouds of carbon atoms, which reduce the XRD peak width by lowering the crystal lattice distortion energy, provide microstructure stabilization and the potential for improved surface fatigue behavior.

Segregation reactions are generally exothermic. The data in Fig. 3b suggests an inverse course of the apparent saturation levels of the surface peak width decrease with rising reheating temperature. The overall kinetics of the complex process may be controlled by the availability of carbon atoms in the matrix or their diffusion to the dislocation cores. The latter is excluded by an estimation based on the Einstein equation, \( \frac{x^2}{t_d} = 2D \). Calculating the mean diffusion distance \( x \) from the conservatively estimated dislocation density \( \rho = 10^{11} \text{ cm}^{-2} \) in the most deformed surface zone, \( x = \frac{1}{2} \sqrt{2D} \).

Fig. 5. TEM image of temper carbides in LT material.
and applying the extrapolated carbon diffusivity $D$ in ferrite [11], which is similar to tempered martensite, one obtains $t_d < 0.5$ s for the diffusion time at a reheating temperature of 180 °C. According to Figs. 1b and 3a, the required duration for reaching half the apparent saturation level of the surface peak width change is three orders of magnitude longer. Assuming that the line width reduction is proportional to the number of atoms segregated at dislocations, rate-controlling carbon release by carbide dissolution according to a first-order reaction law, $\partial m/\partial t \propto -k \times m$, yields:

$$\Delta \text{FWHM}_{z=0}^{\text{TSSA}}(t) = \Delta \text{FWHM}_{z=0}^{\text{sat}} \times [1 - \exp(-kt)]$$

(1)

This relationship describes the PMTT effect with saturation (sat) at $t \to \infty$. Here, $m$ and $t$ stand for the carbide mass and time, respectively. The reaction constant $k$ meets an Arrhenius expression with pre-factor $k_0$, activation enthalpy $\Delta H$, universal gas constant $R$, and absolute temperature $T$:

$$k = k_0 \times \exp\left(-\frac{\Delta H}{RT}\right)$$

(2)

**SIMULATION-AIDED EVALUATION OF THE SURFACE PEAK WIDTH DECREASE**

In Figs. 1a, 1b, 3a, 4a and 4b, the curved lines represent best data fits according to Eq. (1). The derived reaction constants are given. The $k$ values are plotted against the reciprocal temperature in Figs. 6a and 6b, according to Eq. (2). The reaction constant increases with rising reheating temperature. The small deviation between low and high initial surface line widths for LT material may stem from different dislocation densities after finishing. The activation enthalpy below the tempering temperature is comparable with carbon diffusion in ferrite (90 kJ/mol) but markedly lower than for iron self-diffusion (270 kJ/mol) [10, 11], which occurs much slower than the PMTT effect. Since temper carbides in HT material are larger and more stable, dissolution is decelerated, as in bainite [5]. Above the tempering temperature $T_{\text{temp}}$ of 240 °C, where a jump of the reaction constant appears, retempering and thermal recovery further decrease the peak width.

**EXTENSION OF THE PMTT MODEL BY THERMAL DISLOCATION RECOVERY**

By considering the kinetics of static (thermal) recovery of dislocation by an Avrami equation [10]
\[ \Delta FWHM_{\text{Recovery}}(t) = FWHM_{z=0}(t = 0) \times \{ \exp[-(k' t)^b] - 1 \} \]  

(3)

the basic model for the PMTT effect in Eq. (1) is extended to higher reheating temperatures:

\[ \Delta FWHM_{\text{PMTT}}(t) = \Delta FWHM_{\text{TSSA}}(t) + \Delta FWHM_{\text{Recovery}}(t) \]  

(4)

The material constant \( b \) is found to be around 0.4. For \( k' \), an Arrhenius expression according to Eq. (2) with parameters \( k_0' \) and \( \Delta H' \) is valid. The results of prolonged reheating experiments on material HT can be described quantitatively by the extended fitting function in Eq. (4) as shown in Fig. 7, cf. Fig. 3a. As thermal static strain aging occurs much faster than recovery, the effects are clearly separable. In Fig. 7d, where the derived \( k_0' \) and \( \Delta H' \) values are given, this refined data evaluation reveals a TSSA saturation level of 0.27° at higher reheating temperatures. The reaction constant \( k \) at 280 °C is increased by around 15 %, whereas it does not change at 180 and 230 °C.

\[ \begin{array}{c}
\text{HT (tempered at 240 °C for 2 h)} \\
\text{PMTT at 180 °C} \hspace{1cm} \text{PMTT at 230 °C} \hspace{1cm} \text{PMTT at 280 °C}
\end{array} \]

\[ \begin{array}{c}
\text{HT (tempered at 240 °C for 2 h)} \\
\text{TSSA + Recovery} \hspace{1cm} \text{TSSA + Recovery} \hspace{1cm} \text{TSSA + Recovery}
\end{array} \]

Figs. 7a to 7d. Prolonged reheating of material HT: a) 180 °C. b) 230 °C. c) 280 °C. d) Comparison of data evaluation according to Eq. (1), cf. Fig. 3a, and Eq. (4).

ACCELERATED ROLLING CONTACT FATIGUE TESTS

RCF tests under poor lubrication are carried out on surface finished balls made of LT material to determine the effect of reheating at 150 °C for 30 min, which results in a surface XRD line width
reduction of about 0.23°. A significant increase of the $L_{50}$ life at 50 % failure (spalling) probability, estimated from the derived Weibull curves, by a factor of two from 138 to 278 min is found. Simple air reheating may thus improve the surface fatigue and wear behavior of Hertzian loaded parts like bearings, cogwheels and camshafts. PMTT in nitrogen inhibits a nanoscale oxide layer.

OUTLOOK: REHEATING AFTER COLD WORKING

The PMTT effect also occurs after cold working. As an example, Figs. 8a and 8b present results of 190 °C reheating of rumbling (i.e. inverse shot peening) treated rollers. After 1 h, small thermal reduction of compressive residual stresses is accompanied by a marked decrease of the line width in the dislocation-enriched zone by more than 0.2°, which is attributed to thermal static strain aging. The secondary $FWHM$ increase after 1 week of reheating is to be investigated in the future.

Fig. 8a, 8b. Reheating of cold worked SAE 52100. The depth profiles are recorded by stepwise electropolishing at different positions: a) Residual stresses. b) XRD peak width.

REFERENCES