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CHARACTERIZATION OF THE DEFECT DEPTH PROFILE OF SHOT PEENED STEELS BY TRANSMISSION ELECTRON MICROSCOPY

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ABSTRACT

Cylindrical specimens of the plain carbon steel SAE 1045 and the austenitic steel AISI 304 were shot peened under similar conditions. The resulting properties of near surface regions were characterized in a conventional way using X-ray diffraction techniques and micro-hardness measurements. In addition, a special cross sectioning technique was applied for transmission electron microscopy (TEM) preparation. The aim of the TEM-analysis was to investigate depth profiles of dislocation configurations. For that purpose, characteristic parameters of the dislocation arrangements such as dislocation densities or cell respective subgrain diameters were quantitatively determined.The results of these observations were correlated with micro-hardness and residual stress depth profiles. This allowed a detailed discussion of the strengthening mechanisms.
Furthermore, shot peened specimens were fatigued up to certain numbers of cycles. A characteristic cyclic hardening/softening behaviour of the specimens was observed, connected with microstructural alterations and severe effects on residual stress profiles.

KEYWORDS

SAE 1045, AISI 304, residual stresses, X-ray diffraction, cross-sectioning, TEM, dislocation arrangements, fatigue

INTRODUCTION

The significance of shot-peening as an outstanding surface finishing treatment has risen to even greater importance with the advancement of new analysis methods in metal physics and materials science during the past decades. Typical characteristics of shot peened surfaces are compressive residual stress maxima and extremely high dislocation densities below the surface resulting from inhomogeneous plastic deformations. In some cases phase transformations occur, leading to additional surface hardening. These microstructural features are generally considered to be the reason for inhibited crack initiation and propagation in components which are cyclically loaded, submitted to corrosive environment or wear.

In practical applications, especially in quality control, it is sufficient to measure the micro-hardness of the shot peened component or, if an X-ray diffraction unit is available, the residual stresses at and beneath the surface.

Consequently, the matter of interest in many hitherto published papers about shot peening are residual stress profiles, half width values of X-ray interference lines, micro-hardness profiles and, of course, the extend of lifetime improvement for different testing conditions. Fewer literature sources deal with residual stress relaxation [1-5] and the cyclic softening/hardening behaviour of shot peened specimens [6].

Only very limited information is available about the dislocation arrangements and densities in shot peened surfaces, determined by TEM [7-9] respective X-ray profile analysis methods [10-14]. However, these investigations are essential in order to understand the involved microstructural mechanisms of hardening or softening in the wake of service load. Therefore a systematic research was started to determine correlations between shot peening conditions, microstructures and residual stresses on one hand and the alterations of these quantities under distinct fatigue loading conditions on the other hand. Characteristic examples of such investigations are presented in this paper for two frequently used steels, namely AISI 304 and SAE 1045 in the as shot peened condition and also additionally cyclically loaded under stress control in tension/compression in the low cycle fatigue (LCF)-regime.

MATERIALS AND EXPERIMENTAL DETAILS

The materials used were the plain carbon steel SAE 1045 (German grade Ck 45) and the stainless austenitic steel AISI 304 (German grade X5 CrNi 18 10). Both were delivered as rolled cylindrical bars with a diameter of 14 mm. The chemical composition of the materials is given in Tables 1 and 2:

Table 1: Chemical composition of SAE 1045

element	C	Si	Mn	P	S	Fe
wt %	0.45	0.21	0.64	0.020	0.029	rest

Table 2: Chemical composition of AISI 304

element	Cr	Ni	C	Si	Mn	S	P	Fe
wt %	18.11	8.57	0.056	0.35	0.59	0.023	0.022	rest

The specimen geometry used is shown in Fig. 1. The cylindrical specimens were turned and two flat planes on opposite sides of the cylindrical gauge length were close-tolerance milled to facilitate microstructural preparation of the near surface regions for TEM-investigations. Moreover, this geometry turned out to be most suitable for the application of strain gauges.

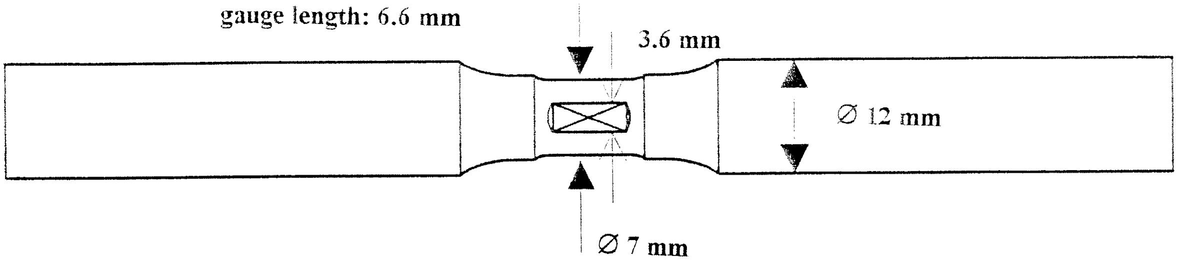


Figure 1: Specimen geometry used

SAE 1045 specimens were stress relieved at 650°C for 1.5 hours in an artificial atmosphere furnace. The ferrite grain size after this treatment was determined by stereological methods as 20 µm. Tensile tests of the specimens revealed the following properties: Upper Yield Strength 478 MPa, Lower Yield Strength 455 MPa, Ultimate Strength 738 MPa, Elongation 22 %. Specimens were shot peened using S170R with a coverage of 98 % and an Almen-intensity of 0.175 mmA.
AISI 304 specimens were not submitted to any annealing treatment, but investigated as delivered. The average grain size was determined as 150 µm (without twins) or 122 µm (including twins). Tensile tests delivered the following mechanical data: Yield Strength 245 MPa, Ultimate Strength 650 MPa, Elongation 62 %. The specimens were shot peened using S170R and a coverage of 98 % with an Almen-intensity of 0.120 mmA. These specimens were subsequently glass-peened to avoid local corrosion.
Tension/compression fatigue tests were performed on a servohydraulic test system with a stress amplitude of 450 MPa for SAE 1045 and 320 MPa for AISI 304 with zero mean stress. Residual stress- and TEM-investigations were carried out on the initial shot peened states as well as on states fatigued up to half the number of cycles to failure. Cyclic softening/hardening curves were obtained by the aid of extensometers and strain gauges.

The residual stress depth profiles were measured with a Ψ- diffraction unit, using the classical $\sin^2\Psi$-method and the gravity line method for peak-determination. The registered peak was the (211)-interference line of ferrite for SAE 1045 and the (220) of austenite for AISI 304. All experiments were performed with chromium radiation with a wavelength of $2{,}2897 \times 10^{-10}$ m. X-ray elastic constants were $\frac{1}{2}\, s_2 = 6.09 \times 10^{-6}$ mm^2/N for SAE 1045 and 6.05×10^{-6} mm^2/N for AISI 304.

Due to the large grain size of AISI 304 it was necessary to use an additional oscillation device to improve the distribution of the diffracted X-ray intensity. Successive removal of material was achieved by electropolishing to determine residual stress depth distributions.

A special preparation method for producing TEM-cross-sections of near surface regions of the specimens was applied (see also [15]). At first, from the specimens the shot peened surface regions were prepared by cutting with a diamond saw. Then two pieces of the specimen were bonded face to face. After that discs for TEM-analysis were prepared by cutting, planar grinding and a final concave dimple grinding. The final ion thinning was carried out by ion polishing under a very small angle of incidence of nearly 4°. Dislocation densities were measured by a computer aided linear intersection method, including a direct estimation of foil thickness, described in [16]. The steps for this special cross sectioning technique are summarized in Fig. 2.

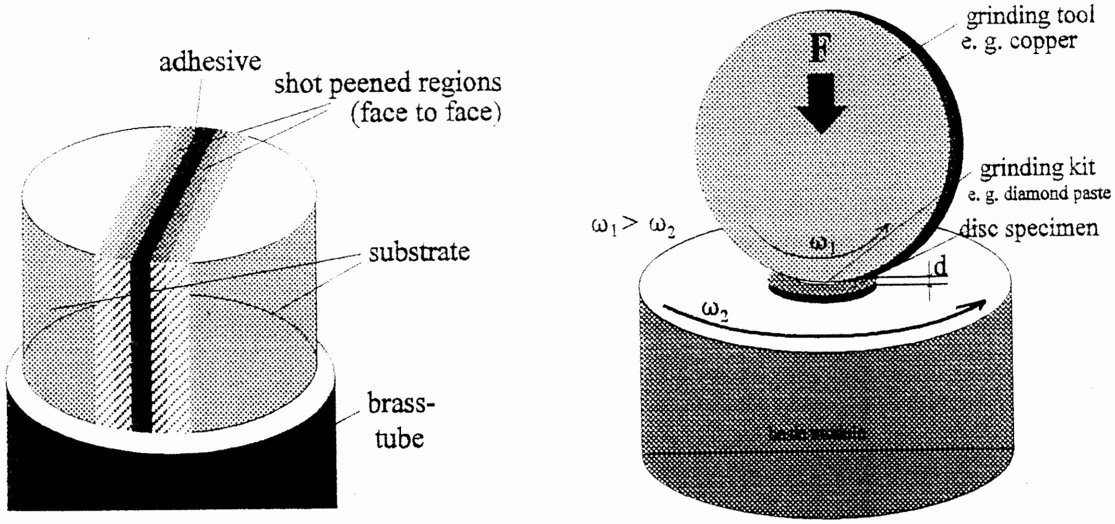
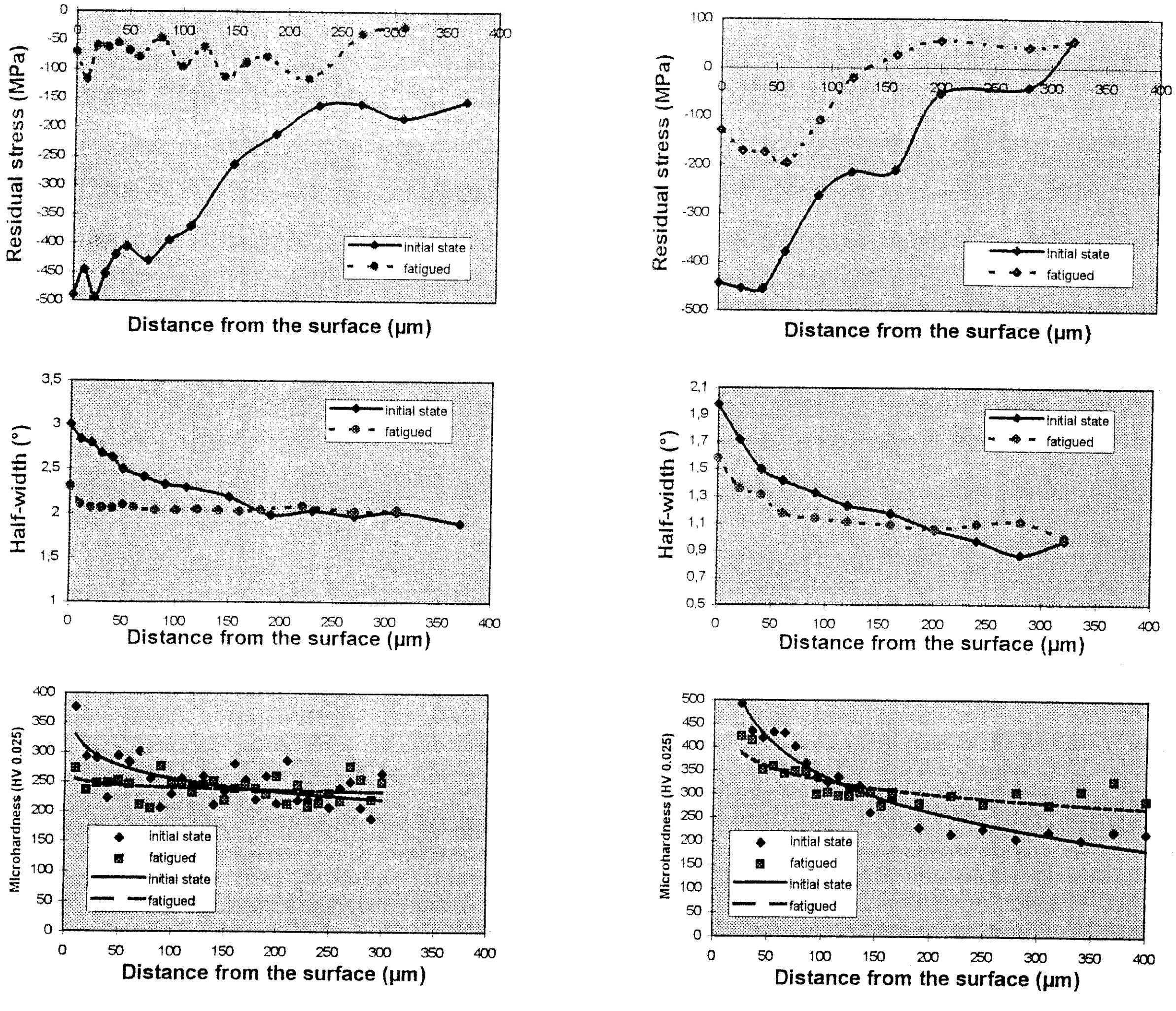


Fig. 2: Steps of the cross-section TEM-preparation: a) adhesive tube technique, b) disc dimple grinding

RESULTS AND DISCUSSION

The measured residual stress depth profiles, interference line half-width values and micro-hardness profiles for both shot peened steels before and after cyclic loading are shown in Fig. 3. The initial shot peened state of SAE 1045 is characterized by a typical residual stress depth profile [17]. Maximum compressive



SAE 1045 **AISI 304**

Fig. 3. Residual stress depth profiles, interference line half-width values and micro-hardness profiles. Left: SAE 1045, Right: AISI 304

SAE 1045

AISI 304

Fig. 4: TEM-defect depth profile of shot peened SAE 1045 and AISI 304

residual stresses of approx. -500 MPa were found directly at the surface, constantly declining up to a depth of approx. 230 µm, where residual stresses of approx. -170 MPa were measured. To greater depths, residual stresses decrease only slightly. Interference line half-width values of the initial shot peened state show a similar development as a function of depth: A maximum half-width of 3° was found in the surface of the specimen, also declining up to a depth of approx. 200 µm down to approx. 2°.

The residual stress depth distribution of steel AISI 304 is similar to that of the ferritic steel. In this case, maximum compressive residual stresses of about -460 MPa were observed in a surface distance of approximately 40 µm. Then residual stress values decrease continuously. As a consequence of the large grain size of the material investigated, a remarkable fluctuation of measured residual stress values was observed for surface distances greater than 200 µm. Interference line half-width values decrease continuously, starting from 1.95° at the very surface to 0.9° in surface distances not affected by the shot peening process.

In principle, the micro-hardness profiles are similar to the half-width value profile, although microstructural interpretation of this parameter is known to be more difficult and less direct than in the case of half-width values [17].

A more thorough interpretation of the involved microstructural mechanisms connected with shot peening is possible with TEM-investigations. Fig. 4 shows TEM-micrographs of the initial shot peened state for both steels. All figures represent cross-sections of the direct or near surface regions. Furthermore, a dislocation density profile of SAE 1045 can be seen in Fig. 5

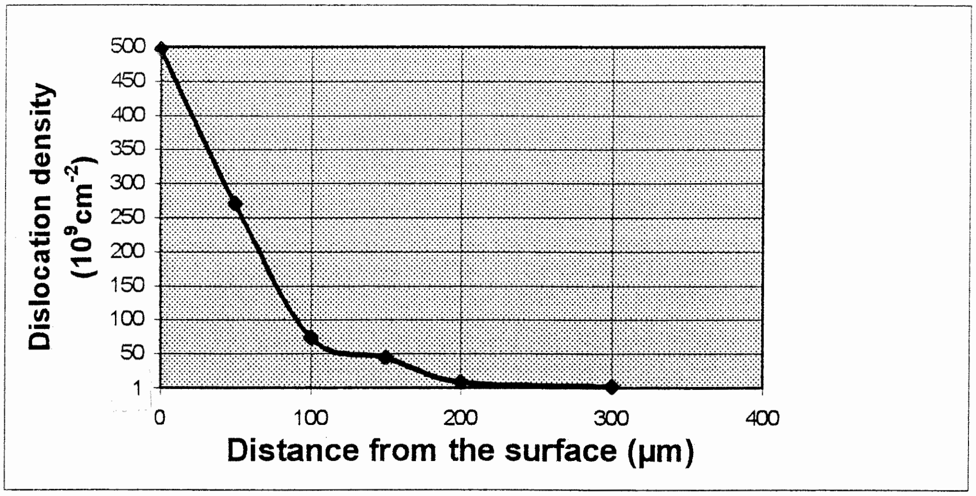
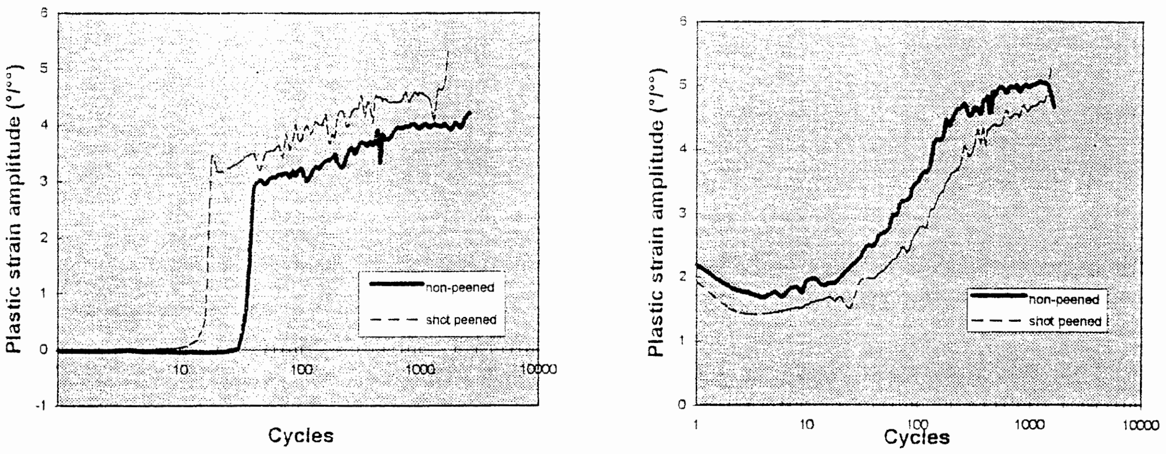


Fig. 5: Dislocation density depth profile of shot peened SAE 1045

TEM-diffraction contrast analysis indicates that in SAE 1045 the direct surface regions exhibit an extremely high dislocation density (5 x 10^{11} cm^{-2}) connected with the occurence of bending contours indicating the high residual stress.

Different dislocation microstructures are present in shot peened AISI 304: Martensitic twin lamellas caused by the shot peening treatment and a dense dislocation network in the austenitic phase can be observed.

The cyclic deformation behaviour of peened and non-peened SAE 1045 as well as AISI 304 can be seen in Fig. 6. During the first 30 cycles non-peened SAE 1045 shows quasi-elastic behaviour. This is usually explained by the building up of a critical dislocation density necessary for plastic straining, therefore this time intervall is also called ´incubation´ time. Then cyclic softening occurs and within a few cycles, the plastic strain amplitude increases drastically. A further continuous increase is observed up to fracture of the specimen. In shot peened specimens softening starts earlier due to the enormous dislocation density in outer layers caused by the shot peening process. For the case investigated the plastic strain amplitude of shot peened specimens constantly exceeds the one of non-peened specimens and there is no lifetime improvement by shot peening. On the contrary,



SAE 1045

AISI 304

Fig. 6: Cyclic softening/hardening curves for non-peened and shot peened specimens. Left: SAE 1045, Right: AISI 304

shot peened specimens took 2050 cycles to failure, compared to 2450 cycles to failure for non-peened specimens (the numbers of cycles to failure are average values of 3 specimens). Furthermore, cracks of 30-50 µm length were found in the fatigued shot peened specimens after half the numbers of cycles to failure.

AISI 304 exhibits plastic deformation already for the first loading cycle. During the first 10 cycles the plastic strain amplitude stays nearly constant, then softening until fracture takes place. Here the plastic strain amplitude of shot peened specimens is comparable, but always lower than the one in non-peened specimens, the numbers of cycles until fracture of non-peened and peened specimens are nearly identical.

For both materials cyclic loading is accompanied by characteristic alterations of residual stress depth profiles, half-width value profiles (Fig. 3) and dislocation arrangements (Fig. 7).

In the case of SAE 1045 after cyclic loading residual stresses due to shot peening have entirely disappeared. This drastic stress relaxation is also expressed in the flattening of the half-width value profile down to the undistorted level of approx. 2°, apart from the very near surface where the degradation is incomplete or delayed.

Obviously, stress relaxation also takes place in cyclically loaded AISI 304 specimens. The residual stresses due to shot peening have decreased by more than 50 % after fatigue loading. However, the remaining compressive residual stresses near the surface still reach considerable amounts of up to -200 MPa.

In comparison to the half-width value profile decline of SAE 1045, the half-width value profile of AISI 304 decreases only slightly after cycling and the reduction is restricted to near surface regions. Up to 200 µm distance from the surface half width values were diminished by 0.1°- 0.4°.

Fatigue loading also affects the micro-hardness depth profiles of both steels. The micro-hardness of SAE 1045 is drastically diminished in near surface regions approximately down to a value of the non-peened ground material. Fatigue loading of AISI 304 decreases the microhardness close to the surface, but increases the microhardness in deeper specimen regions.

Again, further information about microstructural mechanisms can be obtained by TEM-investigations (Fig. 7).

In the near surface regions of shot peened and cyclically loaded SAE 1045 dislocation cell structures have been formed inside ferritic grains. The interior of these cells is characterized by a low free dislocation density of approx. 10^9 cm^{-2} offering a logical explanation for relatively small half-width values and the low micro-hardness after fatigue loading.

The lower plastic strain amplitude of shot peened and fatigued AISI 304 compared to non-peened AISI 304 can be explained by the TEM-observation of strain-induced martensite transformation, making dislocation movement more difficult. This observation was also confirmed for tests with a lower stress amplitude of 280

Fig. 7: TEM-defect depth profile of shot peened and cyclically loaded SAE 1045

MPa, however this time a lifetime-improvement between 30 and 100 % was achieved. Moreover, it must be mentioned that even non-peened AISI 304 is able to transform partially into martensite under fatigue load. This process also takes place during ´autofrettage´ of austenitic pressure vessels [18]. Assessing the cyclic deformation behaviour of both steels it must be considered, that the presented cyclic softening/hardening behaviour does also not automatically represent the behaviour of the strengthened outer layers, since they may be too thin to influence the cyclic softening/hardening behaviour of the whole specimen, similar observations were made by [6].

CONCLUSIONS

The initial shot peened state of SAE 1045 and AISI 304 investigated here is characterized by the well-known typical residual stress-, half-width value- and micro-hardness-depth profile.

In the direct surface regions the dislocation arrangement caused by shot peening is defined by a homogeneous and extremely dense dislocation configuration. These structures develop into typical cell-like structures during fatigue loading.

Small half-width values and low micro-hardness are correlated with a low dislocation density inside the cells of the investigated steel SAE 1045 after fatigue loading. Martensitic twinning caused by shot peening and/or fatigue loading impede dislocation movement in the case of AISI 304.

In the investigated load regime lifetime improvement by shot peening could not be found. For SAE 1045 a possible explanation for this behaviour is the quick change of the dislocation arrangement from a dense, tangled into a cell-structure in near surface regions.

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REFERENCES

[1] O. Vöhringer, H. Wohlfahrt, in: Eigenspannungen u. Lastspannungen, Editor: E. Macherauch, V. Hauk, HTM 87 (1982), 144
[2] J. Hoffmann, B. Scholtes, O. Vöhringer, E. Macherauch, in: Proc. 3rd Int. Conf. on Shot Peening, DGM, Oberursel, 1987, 239
[3] J. Bergström, T. Ericsson, in: Proc. 3rd Int. Conf. on Shot Peening, DGM, Oberursel, 1987, 221
[4] B. Eigenmann, V. Schulze, O. Vöhringer, in: Proc. 4th International Conference on Residual Stresses, Baltimore 1994, 598
[5] A. Bignonnet, H.P. Lieurade, L. Picouet, L. Castex, in: Proc. 3rd Int. Conf. on Shot Peening, DGM, Oberursel, 1987, 585
[6] G. Ebenau, Dr.-Ing. thesis , Karlsruhe, 1989
[7] H. Lu, B. Scholtes, E. Macherauch, Mat.-wiss. u. Werkstofftechn. 23 (1992), 388
[8] H. Xiao, Q. Chen, E. Shao, D. Wu, Z. Chen, Z. Wang, Wear 151 (1991), 77
[9] D.Hakimi, C. Servant, L. Castex, Proceed. 2nd Int. Conf. on Shot Peening, The American Shot Peening Society, Paramus, New York, 1984, 249
[10] J.Y. Wang, Y.X. Tan, R.H. Zhu, Proc. 4th Int. Conf. on Shot Peening, Tokyo, 1990, 191
[11] B. Scholtes, O. Vöhringer, in: Mechanische Oberflächenbehandlung, DGM, Oberursel 1989, 3
[12] O. Vöhringer, in: Proc. 3rd Int. Conf. on Shot Peening, DGM, Oberursel, 1987, 185
[13] G. Maeder, J.L. Lebrun, A. Diament, in: Proc. First Int. Conf. on Shot Peening, Pergamon Press, Oxford, 1982, 263
[14] H. Oettel, Exp. Technik d. Physik 23 (1973), 99
[15] V. Klemm, U. Martin et al. , Prakt. Metallogr. (1996), in press
[16] U. Martin, U. Mühle, H. Oettel, Prakt. Metallogr. 32 (1995), 467
[17] B. Scholtes, Eigenspannungen in mechanisch randschichtverformten Werkstoffzuständen, DGM Informationsgesellschaft, Oberursel, 1990
[18] H.J. Maier, B. Donth, M. Bayerlein, H. Mughrabi, B. Meier, M. Kesten, Z. Metallkunde 84 (1993), 820