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Microstructure of austenitic steel after the oscillatory compression test

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The paper presents the influence of oscillatory torsion on the microstructure of compressed cylindrical samples of austenitic steel. The austenitic steel was deformed at room temperature using a standard compression test and oscillatory torsion test at constant parameters: the torsion angle $\alpha = \pm 5$ and torsion frequency $f_{sk} = 1.8$ Hz. Samples were deformed at strains of $\varepsilon = 4$. Optical observations showed that well-defined slip bands and clusters of slip lines were present in the austenitic steel after two modes of deformation. Specimens deformed during the oscillatory compression test exhibit well-defined deformation twins intersected by shear bands. The structure of shear bands consists of grains between 50 nm and 400 nm in diameter. The analysis of selected diffraction pattern areas has shown that diffraction spots are arranged in rings, indicating that disorientation angles of grain boundaries are higher than 15°. Our investigations constitute a preliminary stage of complex analysis of reactions of metals under diversified process conditions.

Key words: *oscillatory compression test; austenitic steel; substructure; high-angle boundaries*

1. Introduction

Nanocrystalline materials of grain sizes not exceeding 100 nm possess competitive mechanical properties when compared to conventional alloys, and demonstrate much better plastic properties, temperature stability, and corrosion resistance [1]. One of the techniques for producing nanocrystalline materials is the so-called “top-down” method, focused on structure size reduction from the micrometric to nanometric scale. Size reduction may be performed by deformation methods, which allow considerable deformations without losing coherence. Acknowledged plastic deformation methods, which yield nanometric structures include:

- cyclic extrusion compression [2],
- equal channel angular extrusion [3],

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- high pressure torsion methods [1, 4],
- cumulated plastic deformation [5].

New, unconventional deformation methods such as compression with oscillatory torsion have also been implemented. A device for this purpose was developed by Grosman and Pawlicki [6]. The method offers a lot of possibilities with regard to considerable plastic deformations. Control over the process allows one to adjust the compression rate, torsion frequency, and the amplitude of the torsion angle. The paper presents the influence of compression with oscillatory torsion on the structure of austenitic steel. The results obtained indicate that areas characteristic of nanometric structures are formed.

2. Materials and methods

Hot-rolled bars made of AISI304 austenitic steel with 0.03% C, 18.5% Cr, and 8.9% Ni content were used for specimen preparation. Before plastic deformation, for the purpose of dissolving carbide precipitations and obtaining a uniform austenite structure, the samples were subject to solution heat treatment at the temperature of 1150 °C for 60 minutes. The size of austenite grains after thermal treatment was 60 µm.

The research was conducted by a conventional compression method as well as by applying compression with simultaneous oscillatory torsion under conditions of free radial metal flow, using samples in the form of cylinders with initial dimensions of $\phi 10\times 15$ mm^2. The torsion angle amounted to ±5°, and the fluctuation frequency for the lower punch was 1.8 Hz. Based on the recorded data, the relationship between average unit pressure and a true draft was determined.

The structures and substructures of the samples after deformation were examined in the centre of their section height, at the distance of the 0.8 external radius of the sample. The examination of the substructure was carried out with a JEM 3010 high resolution transmission electron microscope operating at 300 kV accelerating voltage and by a JEOL 100B transmission microscope with an accelerating voltage of 100 kV.

The grain sizes were measured with a MOP-AMO 3 semi-automatic image analyser using images obtained from the dark field. The measurements were then used to determine the average equivalent diameter (d) as a measure of grain size as well as frequency distributions for the occurrence of grains of specified sizes. By means of diffraction examinations, the value of disorientation between particular crystallites was determined.

3. Results

The influence of the deformation route on the value of mean unit pressure p_{av} and on the structure of the investigated steel is presented in Fig. 1. The application of compression by oscillatory torsion causes a remarkable decrease in unit pressure values, compared to conventional compression at any phase of the process. The influence of the deformation route enabled cumulating deformation $\varepsilon = 4$ in the material, which has a significant influence on the structure of the steel. In the samples compressed by conventional methods, wide deformation bands were revealed (Fig. 1a). After combined deformation, deformation bands as well as shear microbands can be observed in the steel structure (Fig. 1b).

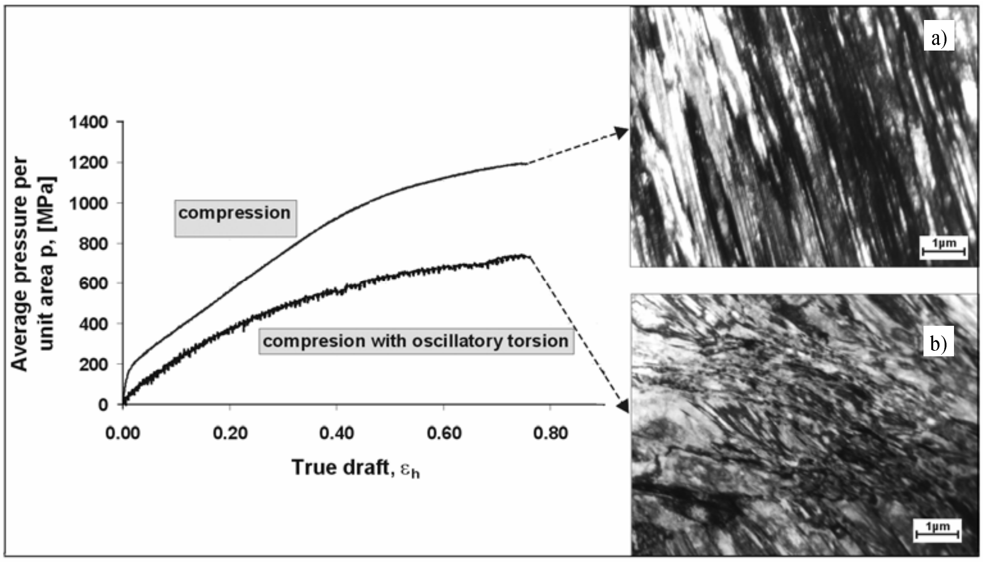

Fig. 1. Influence of conventional compression and compression by oscillatory torsion on mean unit pressure and the structure of the examined austenitic steel

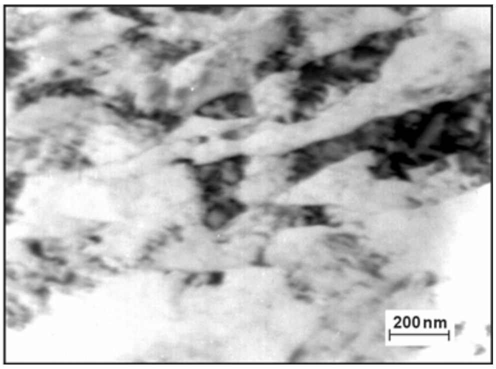

Fig. 2. Nanogranular substructure of austenitic steel after oscillatory compression

A typical substructure created during compression by oscillatory torsion is a band structure running through a number of grains, which indicates that the bands are shear bands. Apart from structures with diverse dislocation densities and indistinct boundaries, structures formed of elongated subgrains occur most often (Fig. 2). Certain areas are characterised by insignificant dislocation densities inside the subgrains. Such a phenomenon is related to structure reconstruction which takes place when considerable deformations are applied. Diffraction patterns obtained from areas of subgrains formed in this way indicate the occurrence of high angle boundaries (Fig. 3).

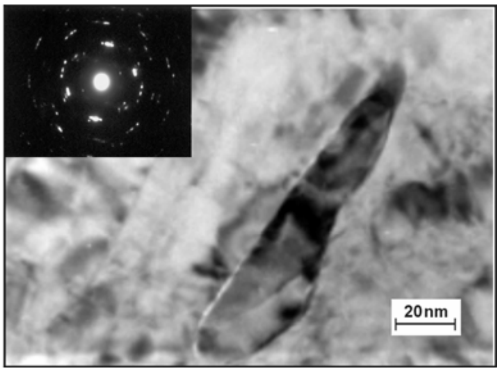

Fig. 3. The area from Fig. 2 in a bright field. Elongated subgrains are visible, with some areas of a reduced defect density. The disorientation calculated from the diffraction pattern amounts to 19°

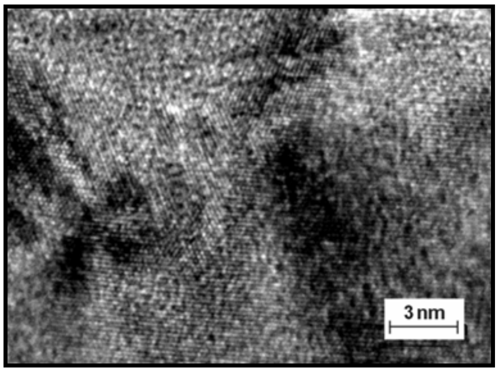

Fig. 4. High-resolution image of the granular structure formed within bands as seen in Fig. 2

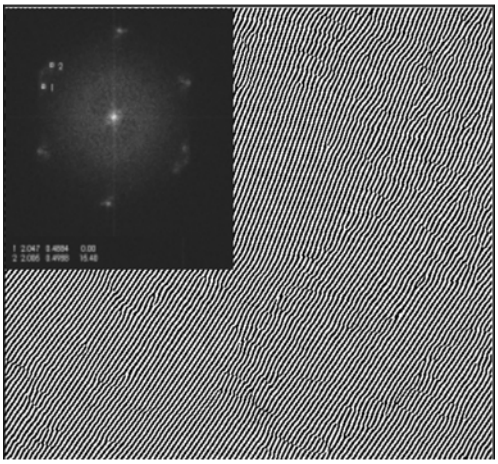

Fig. 5. The area of Fig. 4, with granular structures visible after applying Fourier filtration and diffraction

Based on HREM examinations, it was demonstrated that the structures formed (Figs. 3 and 4) are separated from one another by high angle boundaries (Fig. 4). The value of disorientation presented in Fig. 5, determined on the basis of diffraction examinations, amounts to about 15.5°. It can be expected that a larger deformation will result in a higher misorientation.

The quantitative analysis carried out on the basis of images recorded in the dark field mode has demonstrated that the average diameter of the granular structures formed is 130 nm, with about 70% of the values being in the range 50–200 nm (Fig. 6). The rest of the population is in the range 300–400 nm.

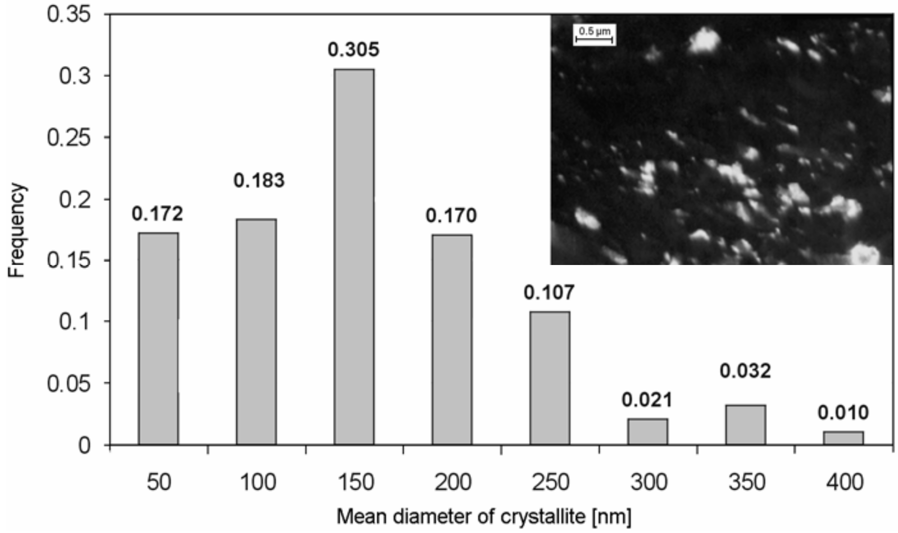

Fig. 6. Frequency distribution of the occurrence of austenitic steel crystallites in specified size classes (equivalent diameters *d*), and an example structure after compression by oscillatory torsion used for the quantitative examination of crystallite sizes, dark field

4. Conclusions

The examinations carried out enabled observation of an intensive influence of the deformation route on parameters related to power and energy and on the structure of the examined steel. Compression with simultaneous oscillatory torsion leads to a considerable decrease in the plastic flow resistance of the metal. The development of shear microbands in the areas of deformation bands is also observed. Within shear microbands, a granular structure is formed, with an average crystallite size of 130 nm. Large disorientation angles (above 15°) between the structures formed, have been found. The share of large disorientation angles between grain boundaries may increase the deformation growth. This can be obtained by a proper choice of process parameters, i.e. the compression rate and the number of oscillatory torsion cycles. The data obtained indicate the possibility of producing nanomaterials from austenitic steel using the method of compression by oscillatory torsion.

References

[1] VALIEV R.Z., ISLAMGALIEV R.K., Progress Mater. Sci., 45 (2000), 106.
[2] RICHERT J. AND RICHERT M., Aluminium, 62 (1986), 604.

[3] SEGAL V.M., Mat. Sci. Eng., A338 (2002), 331.
[4] ZHILYAEV A.P., Acta Mater., 51 (2003), 753.
[5] FERGUSON D., CHEN W., KUZIAK R., ZAJĄC S., Proc. Conf. Mat 5th Int. ESAFORM Conference on Materials Forming, M. Pietrzyk (Ed.), Cracow (2002), 599.
[6] GROSMAN F., PAWLICKI J., Acta Metal. Slov. R, 8 (2002), 178.

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