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Grain size evaluation in strained steels using image analysis*

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In this paper we evaluate the grain size in two austenitic steels after annealing and forming operations. The same micrographs are evaluated both manually and by means of macro program in the software package Lucia. Accuracy of measurement is discussed and suitability of the computer evaluation is commented. In addition, the grain size arrived from metallographic samples of strained steel is compared with the results of X-ray diffraction analysis.

1. INTRODUCTION

Grain size of polycrystal materials has considerable influence on mechanical properties and its measurement belongs to common tasks of metallographic evaluation. The explosion of computer technologies coupled with digital-image acquisition devices and progress in methods of image analysis make possible to replace the time-consuming manual measurements by computer evaluation.

If we want to determine the mean grain area, the mean linear grain size or the grain boundary density, we have to count grains in defined area or along defined lines. The number of grains we have to put into well-known relations [1]. In the case of computer evaluation, image processing has to be performed before these calculations [2]. The sufficient sequence of the image operations must be specified and the user should choose the proper parameters of selected operations, because inappropriate image editing can result in a large measurement error.

The grain size is solely measured in metallographic samples using light microscopy. Only those grain boundaries can be included in evaluation, which have been etched. However it is in question, if boundaries observed in microscope correspond with real grains. That is why the results of X-ray diffraction analysis are also included in this paper.

2. EXPERIMENTAL PROCEDURES

The grain size measurements of three types of austenitic structure (annealed, cold strained and hot strained structures) were performed both manually and using computer evaluation.

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Cr-Mn-N steel (0.06 C, 19 Cr, 18Mn, 0.5N) and Cr-Ni steel (0.07 C, 2 Mn, 18 Cr, 10 Ni) were used for investigation. Chemical composition is given in wt.%.

Two sets of specimens were machined out from Cr-Mn-N steel. Specimens of the first set were processed in different methods of thermo-mechanical processing with annealing at 1030°C for 2h as the last operation. Specimens of the second set were annealed at 1030°C for 2h and cold strained at compression deformation. The third set of roll-shaped specimens made from Cr-Ni steel was preheated at 1100°C for 1h and lengthened between flat swages using 1, 3, 6 or 9 compression deformations with specimen rotation of 90° after each deformation. The total deformation was about 50%. Metallograpfic samples were etched in order to reveal grain boundaries.

For manual assessment the three-circle method was used and mean grain size was calculated from the equation $d_m = k.\ D/a$, where k is a constant, D is the circle diameter and n is the number of intersections. Constant k is derived on conditions that grains are globular and very small. For random grain sections k was assumed to be 4 [3].

For computer evaluation a special macro program was created in IA software package Lucia 4.6. On the very beginning variables are specified. Then image-processing follows consisting of automatic operations and some selected reversible operations controlled by the user. From modified binary image equivalent diameter is determined and written into the table. Equivalent diameter is an attribute of size derived from area and specifies diameter of circle, which has the same area as corresponding object [2]. If correction of random grain sections is taken into account, then mean grain diameter is given by $d_c = 1.27\ d_e$.

X-ray diffraction analysis was carried out for hot strained specimens using a cobalt anode and the photo registration in Bragg-Brentan semifocusing arrangement.

3. RESULTS AND DISCUSSION

The first set of processed and annealed specimens was used for verification of grain size measurement by force of the macro program. Values obtained manually (d_m) were compared with corresponding mean grain diameters (d_c) arrived from computer evaluation. The relative measurement error of grain size does not exceed 10%.

The second set of cold strained specimens served especially for development of sufficient grain boundary etching. The best results were obtained after electrolytic etching in a 60% fresh aqueous solution of nitric acid at a voltage of 1.5V for 100s using the apparatus Lectropol 5. These conditions were used for both steels that were investigated.

The third set of hot strained specimens was prepared in order to extent grain size measurement on strained and recrystallized microstructures. The strain distribution in these specimens is very heterogeneous and consequently different microstructures can be observed in each of metallographic samples. During forming operations recrystallization proceeds and the microstructure becomes finer proportionally to the number of deformations. Structural changes are evident especially in the axial cross-sections of specimens. Two main parts can be distinguished: central field bounded with double contour and rest marginal zone (Fig. 1a). Fine-grained structure dominates within central region (Fig. 1b), while relatively rough structure is near the edges of specimens (Fig. 1c).

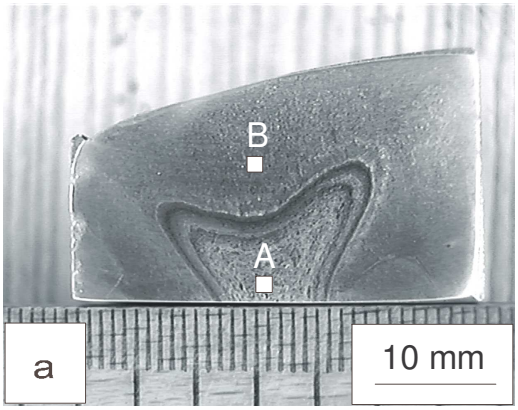
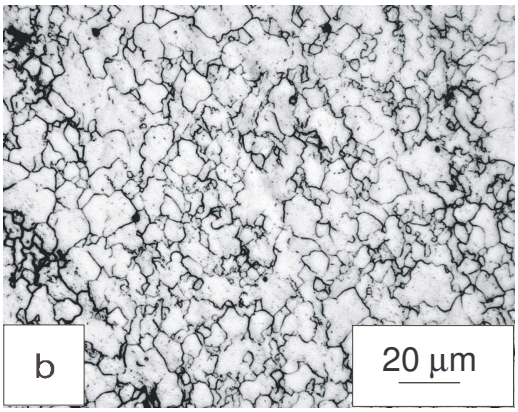
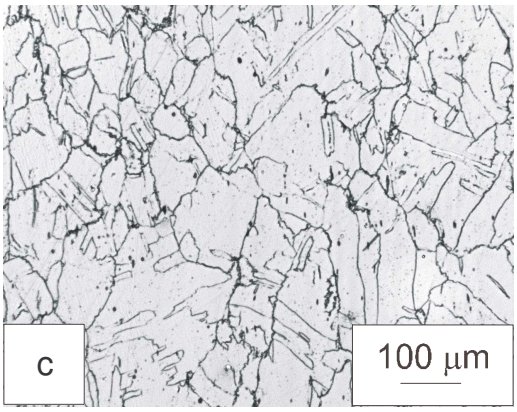


Fig. 1. Specimen 4:
a) macrostructure (etching in solution of 50ml HCl, 10g $CuSO_4$ and 50ml H_2O),
b) microstructure in the A region,
c) microstructure in the B region (electro etching in 60% solution of HNO_3).





Large amount of fine recrystallized grains in a strained matrix can be observed along the dark contours. In each specimen two regions with uniform grain size were chosen for grain size evaluation: the central A region and the B region out of the contours, where no fine recrystallized grains are visible in light microscope. In the same regions X-ray diffraction analysis was performed. From diffraction patterns the size of coherent scattering regions was estimated. The coherent scattering region represents the volume of sample in which only one diffraction spot is originated. If the size of coherent scattering regions is higher than 10μm, diffraction patterns are separated (Fig. 2). If these regions are smaller than 10 μm, diffraction patterns form continual lines [4]. The results of hot strained specimens evaluation are given in Table 1.

Table 1.
The sizes of austenite grains and coherent scattering regions in hot strained Cr-Ni steel

Region	A				B			
Specimen	1	2	3	4	1	2	3	4
Number of deformations	1	3	6	9	1	3	6	9
Mean grain diameter d_m [μm]	30	12	7	6	91	53	32	35
Mean grain diameter d_c [μm]	25	15	8	7	51	37	28	33
Size of coherent scattering regions [μm]	50	40	30	20	<10			

Corresponding results of manual and computer evaluation of hot strained specimens differ more than the results of annealed and cold strained specimens. Especially in the B regions values of d_m differ from d_c. Grain sizes obtained manually are systematically higher than values arrived from computer evaluation. For specimen 1, which was strained at one deformation, it is possible to put the relation $d_m \cong 2d_c$. If the number of deformations increases, mean grain diameter d_m approaches to d_c. This fact is chiefly due to image processing, specifically morphological separation and hand drawing operation, which can be influenced by the user. During these operations fragments of etched grain boundaries are spliced and consequently number of grains in selected area increases.

Comparing the mean grain diameters with the size of coherent scattering regions we can arrive at a new view of microstructural processes. In marginal zones, where recrystallization after last deformation did not take place, the size of coherent scattering regions is smaller than 10 μm, although grain size obtained metallographically represents tens micrometers. This means that compression deformation results in splitting of grains into smaller blocks, which behave as independent grains. In the central zones sizes of coherent scattering regions seem to be larger than grain sizes. This conclusion is surprising and can be explained in the following way. During high temperature exposure recrystallized grains probably overgrow boundaries of primal deformed grains, however their paths are not completely "deleted" and thus they can be etched within metallographic sample. In addition, repeated recrystallization results in structure with irregular shapes of grains. If the plane of metallographic sample intersects adjacent grains near their edges they can be displayed as a group of grains.

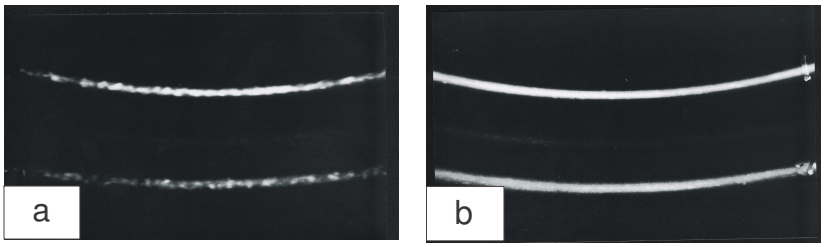


Fig. 2. X-ray diffraction patterns obtained for specimen 3:
a) from the A region,
b) from the B region.

4. CONCLUSIONS

The macro program in IA software package Lucia for grain size evaluation gives sufficient results. It can be used for both annealed and deformed structures. For strained specimens it is recommended to perform also X-ray diffraction analysis and compare sizes of grain with that of coherent scattering regions.

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