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RECRYSTALLIZATION DIAGRAM OF IODIDE ZIRCONIUM

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The size of the grains of a metal or alloy, as is known, largely determines the quality and mechanical properties of the finished product. Therefore, the study of recrystallization processes that relate grain size to the degree of deformation and the annealing temperature is of great practical as well as theoretical interest.

With regard to zirconium, the literature contains some data on the annealing temperatures of zirconium after deformation ([1–3]), but a complete recrystallization diagram for iodide zirconium has not yet been published. We have constructed a recrystallization diagram (of type 1) for the case of annealing cold-deformed iodide zirconium (99.7% Zr).

Rods of iodide zirconium, 16–18 mm in diameter, deposited on a zirconium filament, were taken as the starting material. The hardness of this zirconium was 80–82 kg/mm^2. To obtain a uniform initial structure, the rods of iodide zirconium were subjected to cold rolling deformation with a reduction of 50% and subsequent annealing at 600° for 1 hour. No strong grain growth was observed in this case, although recrystallization was already occurring; the structure was polyhedral and fine-grained, with an average grain diameter of about 15 μ (Fig. 1, *a*). This preliminary sample-preparation regime was chosen as a result of a series of experiments with different degrees of deformation, different temperatures, and different annealing times.

To construct the recrystallization diagram, deformation of the samples prepared in this way was carried out by cold rolling with reductions from 2.5 to 90% at intervals of 10%. At higher degrees of deformation, rolling was performed in several passes, with the sample being deformed by no more than 10% in each pass. With increasing degree of deformation, gradual grain refinement, development of texture, and deformation twins visible in many microstructures were observed. At deformations of 70 and 90%, the microstructures have the appearance of parallel-oriented fibers (Fig. 1, *b*). Annealing of the deformed samples was carried out in evacuated quartz ampoules for 1 hour at temperatures of 500, 550, 600, 700, 800, 900, 1000, and 1200°.

The annealed samples were subjected to microstructural and X-ray analyses, as well as hardness measurements. Polished sections were photographed in re-

Fig. 3. Recrystallization diagram of iodide zirconium

Figure 1: Fig. 3. Recrystallization diagram of iodide zirconium

flected and polarized light. To reveal the structure, various etchants consisting of mixtures of hydrofluoric and nitric acids were used. Grain size was determined by calculating their average diameter under the microscope and on microphotographs.

Annealing of cold-deformed samples substantially affects the size and shape of the grains. All samples annealed in the region of existence of the α-modification of zirconium (up to 800°) had a uniform polyhedral structure (Fig. 1, *v*). Annealing of samples in the β-region (from 900 to 1200°) causes a sharp increase in grain size and a change in its shape (Fig. 1, *g*). The grains acquire—

Fig. 1. Microstructures of iodide zirconium. 200×: *a*—recrystallized zirconium (initial state); *b*—deformed by 90%; *c*—deformed by 40%, annealed at 700°, photographed in polarized light; *d*—deformed by 40%, annealed at 900°, photographed in polarized light.

Fig. 2. X-ray diffraction patterns of iodide zirconium: *a*—initial recrystallized zirconium; *b*—cold-rolled by 7.5%, not annealed; *c*—cold-rolled by 20%, annealed at 550°; *d*—cold-rolled by 5%, annealed at 600°.

have an irregular configuration, and needles of the transformed β-phase can be observed in them. The presence of a needle-like microstructure is also a sign of contamination of zirconium with oxygen (0.03–0.04%). X-ray structural analysis of specimens annealed at 800 and 1300° showed the presence only of the hexagonal structure of α-zirconium. X-ray analysis also made it possible to establish the onset of recrystallization at various temperatures. The Debye-pattern photographs were taken in molybdenum radiation with exposures from 1.5 to 2 hours. Figure 2 shows the X-ray patterns of the initial (a), deformed (b), and annealed specimens (c, d). It was established that, in iodide zirconium, recrystallization begins at 500° in specimens deformed by 50–90%; at 550° in specimens deformed by 7.5%; and at 600° already in all deformed specimens. Hardness was measured with a diamond pyramid under a load of 10 kg. The hardness of the deformed specimens gradually increases with increasing degree of cold deformation. Annealing at 500° noticeably lowers the hardness of strongly deformed specimens. Annealing at 600°, and especially at 900°, substantially reduces the hardness of work-hardened zirconium (Table 1).

Fig. 3. Recrystallization diagram of iodide zirconium

Table 1

Change in the hardness of iodide zirconium as a function of annealing temperature
(H_v, kg/mm^2)

Annealing temperature (°C)	0	10	20	30	40	50	60	70	90
Deformed	82	90	94	100	118	121	–	122	135
500	80	90	94	100	100	103	–	108	–
600	76	64	76	75	78	78	75	79	73
900	67	61	62	67	60	60	65	64	63

On the basis of the experimental data obtained, a recrystallization diagram was constructed for iodide zirconium (Fig. 3). The line of the onset of recrystallization (according to X-ray data) is drawn as a dotted line with crosses. Grain growth at the critical degree of deformation begins to appear from 700°. The "critical" degree of deformation up to 1200° does not exceed 2.5–5%. Noticeable grain growth at all deformations is observed above 800°, i.e., in the β state of zirconium. For example, for 40% deformation, the average grain diameter after annealing at 700° is 18 μ, and after annealing at 900° it is 80 μ. Annealing above 1000° has little effect on grain growth, and the grain sizes at comparatively large deformations differ little from one another.

It is possible that this occurs because of some oxidation of the grain boundaries during annealing in evacuated quartz ampoules. The unusual course of the grain-size isotherms at 800 and 900° is noteworthy; after pro-

Upon reaching the critical degree of deformation, the grain size does not decrease, as it usually does according to a hyperbolic dependence, but, on the contrary, increases with increasing degree of reduction. Therefore there are grounds to suppose that, in the course of isotherms of grain size at high degrees of deformation and high annealing temperatures, a second maximum appears in the purest zirconium. A special check carried out by us showed that the recrystallization diagram of twice-refined iodide zirconium (99.85% Zr) coincides with the recrystallization diagram shown in Fig. 3 for ordinary iodide zirconium.

In conclusion, on the basis of the recrystallization diagram, it may be recommended that annealing of cold-deformed zirconium be carried out at 700–750°. At these temperatures the deformed structure is completely restored (recrystallization treatment), the grain sizes increase only slightly, and zirconium articles are little oxidized at these temperatures even when annealed in air.

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Note: Figure translations are in progress. See original paper for figures.