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Deformation tracks distribution in iridium single crystals under tension

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Abstract The deformation tracks distribution in a single crystal of the high melting FCC-metal iridium, which exhibits the cleavage after considerable elongation, is considered. The octahedral slip is the sole deformation mechanism in iridium single crystal at room temperature, and, therefore, its mechanical behavior is similar to the behavior of a FCC-metal. However, in contrast to other FCC-metals, the resource of plasticity of the iridium single crystalline samples is exhausted at the initial/early stages of plastic deformation, when the octahedral slip bands are homogeneously distributed on the working surface and the necking is absent in vicinity of the dangerous crack.

Introduction

Metals having the face centered cubic (FCC) lattice are considered to be the standard for a highly plastic crystalline solid: strong work hardening does not result from considerable plastic flow of material, especially in the single crystalline state [1–3]. The high mobility of the perfect dislocations with ⟨110⟩ Burgers vectors, which carry the octahedral slip, is the cause for such behavior [4]. There are no strong obstacles for the motion of dislocations at the initial stage of plastic deformation in pure FCC-metals, in contrast with pure BCC-metals where such ones can exist [1, 3, 4]. The fracture mode of a FCC-metal has also been accepted as the standard for ductile fracture [3, 5, 6]. This knowledge, however, applies only to FCC-metals whose melting points do not exceed 1800 °C. The only high melting FCC-metal iridium ($T_{melt} = 2443$ °C) exhibits another type of deformation and fracture behavior. At room temperature, iridium single crystals are highly plastic material, which strengthens severely and, under tensile stress, cleaves [7–10]. However, a formal comparison of plastic deformation of iridium with an FCC-metal, for example, with copper, (in the frameworks of the well-known empirical approach to plastic deformation of metals [3]) has shown their mechanical behaviors are similar[1] [8, 10]. Despite this, some physical properties of iridium and their general agreement with empirical cleavage criteria permit a qualitative indication that cleavage is an intrinsic property of this FCC-metal [11], but no detailed mechanisms of the iridium anomaly have yet been proposed [10–13]. Therefore, an experimental study of mechanisms of plasticity in iridium single crystals under tension is the important step to obtain a better understanding of the cleavage fracture in an FCC metal.

Experimental procedure

The Ekaterinburg Non-Ferrous Metals Processing Plant provided the iridium single crystals for the research on loan. The technology for refining iridium, the technique of growing single crystals and information about the impurity contents in the material were briefly described in [14]. That paper provides the basis for the conclusion that their iridium does not contain dangerous non-metallic impurities such as carbon and oxygen. Samples for mechanical tests (parallelepipeds having the size of 15–20 × 2 × 0.5–1 mm

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[1] See Appendix

with the tensile axes along ⟨100⟩ or ⟨110⟩ and the surfaces of {100} or {110}) were cut from the massive iridium single crystalline ingots by spark erosion. Their back surfaces were electropolished in an aqueous solution of $CaCl_2$ with AC. Tensile tests were carried out at room temperature with a traverse rate of 1 mm/min.Working surfaces of the single crystalline samples were documented in detail using an optical metallographic microscope prior to and after the mechanical test. Topograms were derived by merging the obtained images in a personal computer. The topograms served as the main database for the determination of deformation tracks, their geometry and the distribution on the surface. A transmission electron microscope (TEM) study of the dislocation structure in iridium thin foils was carried out to verify the conclusions.

Results

In the beginning, the mechanical behavior of iridium single crystals under tension will be briefly outlined. Deformation curves of the single crystalline samples are shown in Fig. 1. Their behavior regarding the orientation anisotropy of yield stress (10 MPa for ⟨100⟩ and 100 MPa for ⟨110⟩) and strengthening was considered in [15, 16]. The rectilinear trajectory of the curves for both hard ⟨100⟩ and soft ⟨110⟩ directions should be especially noted; since this feature permits the conclusion that, the whole of the considerable elongation of iridium single crystals prior to failure occurs during the one stage of plastic deformation. The deviation of the curves from the rectilinear shapes in the final of the tensile test may be explained by the appearance of the dangerous (dominant) cracks in the samples. Hence, in contrast to the deformation behavior of the other FCC-metals [17], high melting iridium exhibits both considerable difference between the magnitudes of $\sigma_{0.2}$ and σ_b, 10÷100 MPa and 600 MPa, respectively, and failure (cleavage) in *the initial/early stage* of plastic deformation (inasmuch as no other stages are observed on the tensile curves of the single crystalline samples). At what the word *''the initia/earlyl stage''* does not mean the terms ''stage 1'' or ''easy slip stage'' of plastic deformation in metallic single crystals [3, 17]; it is just an attempt to find an appropriate word. The highest yield stress of iridium single crystals (the magnitude of $\sigma_{0.2}$ ~ 10÷100 MPa) in comparison with other FCC-metals (≤ 10 MPa) may be considered as consequence of the very low dislocation mobility in this FCC-metal [4, 18]. A ''low mobility'' means that applied stress, which is needed for a start of dislocation motion in iridium, is higher than one for other FCC-metals. Taking into account the high melting point of iridium that caused by strong interatomic bonds this suggestion sounds reasonably. Observations of the stable dislocation pileups in the form of networks and braids ahead V-shape microcracks in iridium thin foils (for TEM) are the direct proofs of this conclusion [19, 20]. Besides, any dislocation motions are never detected in TEM experiments (except in situ tension and heating in column of the microscope) that also maintain the supposition.

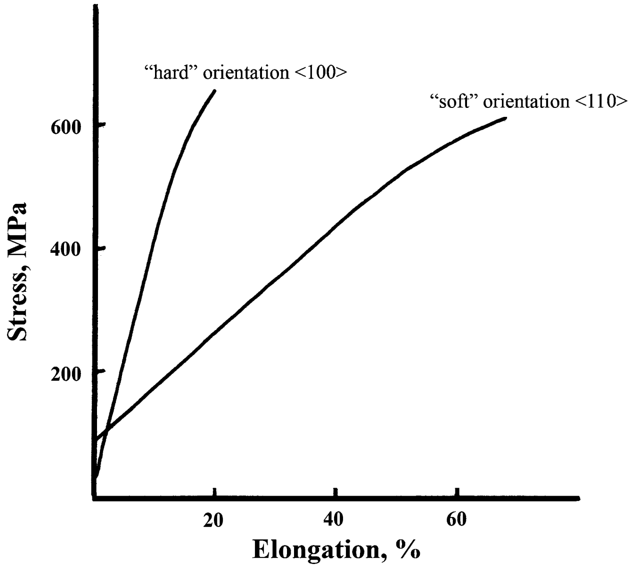

Fig. 1 Deformation curves (tension) of iridium single crystals at room temperatures

The main part of the paper is devoted to distribution of deformation tracks on the tested single crystalline samples. For both hard and soft directions, deformation tracks appeared on the surface of the samples after a total elongation of ~2%. The topogram of the back surface of the sample which had been elongated along the hard (⟨100⟩) direction on the cubic plane is shown in Fig. 2a. The tracks are visible as thin rectilinear lines grouped in the bands that cross over the sample from edge to edge at an angle of 45° to the tensile axis (Fig. 2b). It sounds as a description of the octahedral slip development on the initial stages of plastic deformation in single crystals of FCC-metals [1]. No deformation tracks of any other orientations and shapes are observed in the sample. The wide strips that incline to the tensile axis on 85^o are defects appeared during single crystal growing. It is important to note that they never are a cause of cleavage crack appearance or the lowering of the crystal workability.

The further plastic deformation up to the failure does not cause the changing the character of the distribution of deformation tracks on the back surface (Fig. 2c). Of course, the density of thin lines (the frequency of the individual tracks) in the bands increases with increasing elongation, while no new kinds of deformation tracks appear (Fig. 2d). This is the behavior expected for

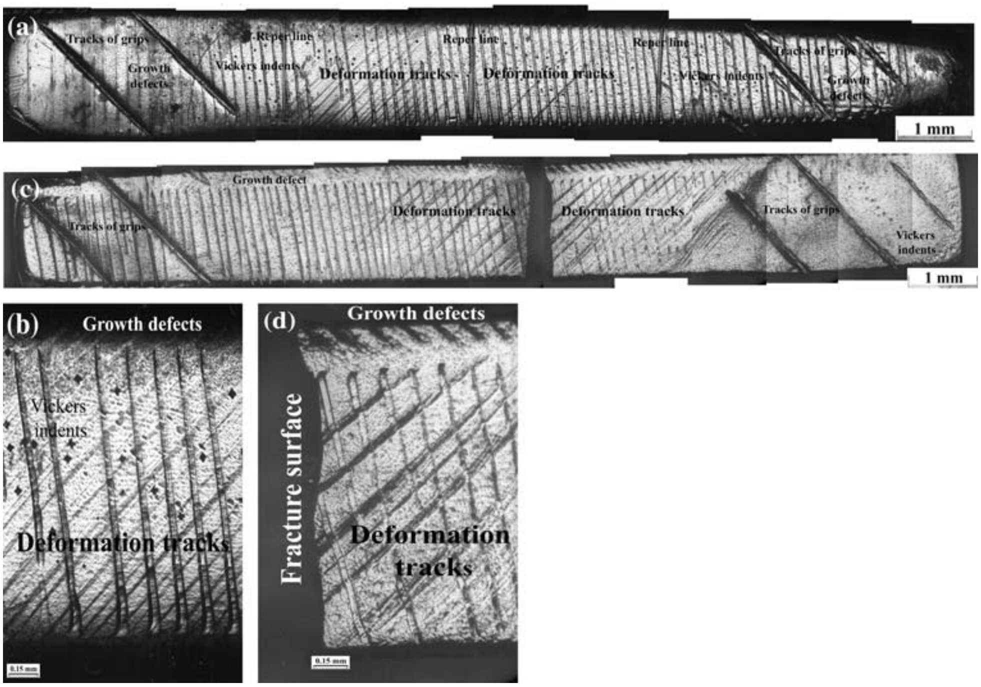

Fig. 2 Deformation tracks on the back surface of iridium single crystals (tension along ⟨100⟩, plane {100}): *a*—topogram of the working surface (elongation 2%); *b*—fragment of the back surface (elongation 2%); *c*—topogram of the working surface (elongation up to failure 10%); *d*—fragment of the back surface (elongation up to failure 10%)

deformation tracks (octahedral slip bands/lines) in a FCC-metal single crystal, which has been elongated along the hard direction [1]. However, no deformation relief and no indications of necking near the dangerous crack are observed on the back surface of the failed sample despite considerable elongation (~10÷20%). Cleavage is the fracture mode of the iridium single crystalline samples having the same orientation (Fig. 3). Thus, according to metallographic observations, there is the sole deformation mechanism in iridium single crystals for this case.

Changing the tensile axis from the hard (⟨100⟩) to the soft (⟨110⟩) direction on the cubic plane {100} leads to the appearance of new features in the distribution of deformation tracks on the working surfaces of the samples, while the shape of the individual tracks remains unchanged. The tracks are homogeneously and equidistantly distributed over the deformed area of the sample (Fig. 4a). These are the thin rectilinear lines, which cross the sample from edge to edge in the normal direction to the tensile axis (Fig. 4b). This behavior also corresponds to the empirical knowledge of octahedral slip in a FCC-metal elongated along the soft direction [1]. There are not other kinds of deformation tracks, features of deformation relief and neck formation on the working surfaces of the samples stretched in the soft direction (elongations prior the failure 30% and more). The fracture mode of the samples is cleavage, too.

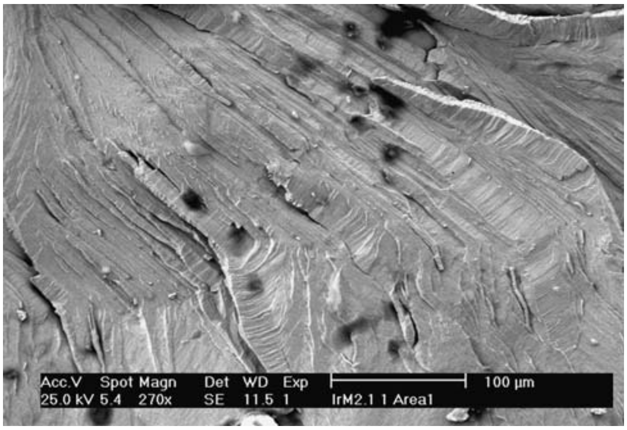

Fig. 3 Fracture surface of the iridium single crystal (tension along ⟨100⟩ axis)

The picture of deformation track evolution on the samples having the same tensile axis but another working surface ({110}) looks similar to described above one. In the initial (undeformed) state any deformation tracks excepted growth defects (marked on Fig. 5a) are absent on the working surface. The tracks, which are thin rectilinear lines, cross over the working surface from edge to edge and cover it homogeneously and equidistantly (Fig. 5b). Their inclination to ⟨110⟩ is close to 35° (Fig. 5c). It should be taken in to an account that the degree of deformation of the sample must be considerable, since its initial geometry, i.e. a parallelepiped, has transformed to a double spoon shape prior the failure (~30% elongation at this photo). No deformation relief and necking in vicinity of the place of failure (cleavage) are also observed.

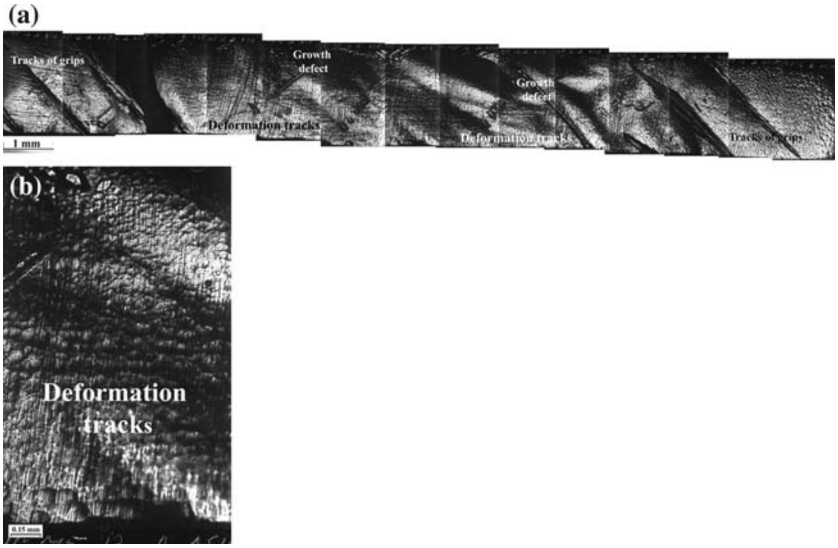

Fig. 4 Deformation tracks on the back surface of iridium single crystals (tension along ⟨110⟩, plane {100}): *a*—topogram of the working surface (elongation up to failure 30%); *b*—fragment of the back surface (elongation up to failure 30%)

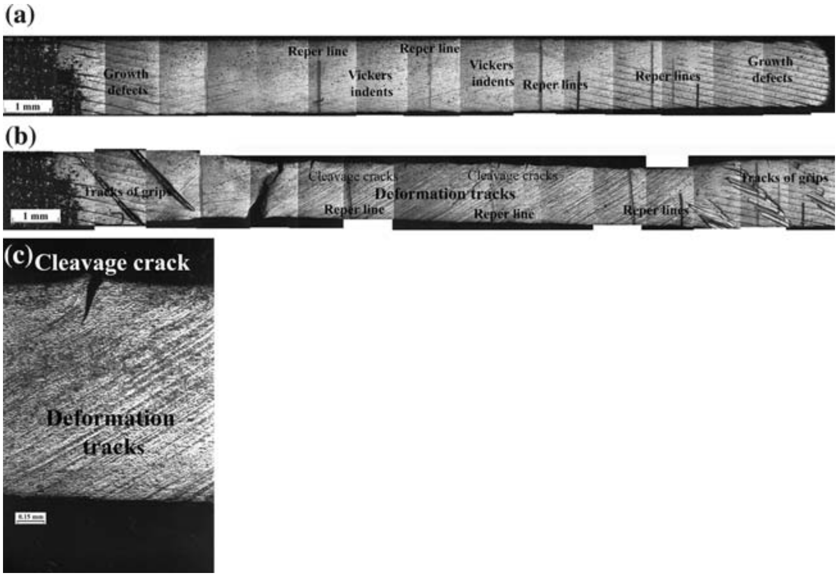

Fig. 5 Deformation tracks on the back surface of iridium single crystals (tension along ⟨110⟩ , plane {110}): *a*—topogram of the working surface (undeformed state); *b*—topogram of the working surface (elongation up to failure 30%); *c*—fragment of the back surface (elongation up to failure 30%)

Discussion

An analysis of the geometry of deformation tracks on tested iridium single crystalline samples allows concluding that they are octahedral slip bands belonged to the one slip system (for every selected crystallographic orientation of sample, of course). No contributions of alternative mechanisms (cross slip, mechanical twinning, non-octahedral slip and so on) to plastic deformation of iridium single crystals under tension at room temperature are detected with a help of a metallographic study. Clear visible octahedral slip bands having the single orientation on the back surface without deformation relief can only occur on the stage 1 and stage 2 (in its beginning) of the plastic deformation of metallic single crystals [1, 3]. Hence, it is reasonably to suppose that all plasticity of iridium single crystals is realized on the initial/early stages of plastic deformation due to octahedral slip. In spite of the high plasticity (elongation of 10÷30%), the sharp V-shape cracks appear on the back surface of the iridium single crystals (Fig. 5c) whose growth causes the cleavage in the samples (Fig. 3).

This conclusion may be verified with a help of TEM attestation of the microstructure in iridium thin foils, since the octahedral slip bands are showings of the processes occurred on the more small (dislocation) scale (level) [3, 21]. Indeed, it is well known what kinds of dislocation structures occur on the initial/early stages of plastic deformation (stage 1 and stage 2) in metallic single crystals: they are single dislocations and dislocation pileups

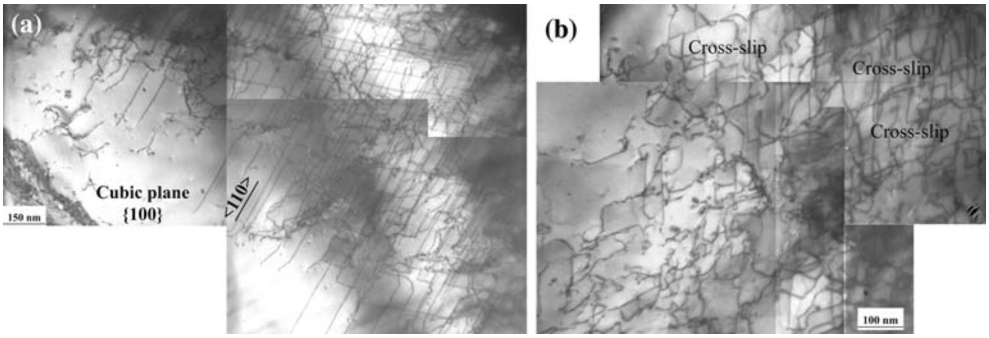

Fig. 6 High dense dislocation networks in the iridium single crystalline thin foil: *a*—networks near the foil edge; *b*—dislocation cross-slip in the networks

including networks [3, 22–24]. TEM study of the single crystalline iridium has shown that the highly dense networks fill the volume of thin foils [16]. Its density is so great that only darkness is observed on the microscope screen, while electronograms including central beam and other spots are clearly visible here. Sometime, the turning of the foil on the big angles allows observing the fragments of these nets on the screen. In addition, the edges of the highly dense dislocation networks could be revealed near the foil edges (see Fig. 6a). Unfortunately, high working magnification (×50 000 and more) and small transparent areas in the foils (due to considerable thickness of material and high atomic weight of iridium) did not allow to carry out detailed crystallographic attestation of dislocations in iridium. However, a primitive contrast analysis of dislocations on cubic plane (see [25]) has been fulfilled and, according to it, dislocations in iridium thin foils have $\langle 110\rangle$ Burgers vectors [26]. This conclusion agrees with the results of the recent work [27]. The nets consist of the long rectilinear dislocation fragments that sometimes (when a primitive attestation was possible) are directed along $\langle 110\rangle$ directions, that was revealed in the first in [28]. The transformation of a network into a cellular structure never occurs in iridium single crystalline thin foils at room temperature. Therefore, the highly dense networks may be considered as the stable and the most advanced dislocation configuration in iridium thin foils under these experimental conditions. Perhaps, this fact is connected with a low mobility of dislocations in iridium at room temperature. No microtwin lamellas are observed as result of the plastic deformation. Hence, the results of TEM study supports the conclusion that all plasticity of iridium single crystals under tension is realized on the initial/early stages of plastic deformation.

The question about the causes of stability of the dislocation networks that directly connected with the iridium problem arises. According to Fig. 6a, both intersection of $\langle 110\rangle$ dislocations and their termination on the crystallite boundaries may be considered as the mechanisms for the highly dense network stabilization (if a task is formulated for the FCC-metal in the frames of the dislocation theory). On the other hand, the cross-slip of $\langle 110\rangle$ dislocations, may be also taken into account, since sometimes it occurs in iridium thin foils (Fig. 6b). Indeed, the first principal calculations of the defect structure in iridium point to an important role of dislocation cross-slip in the deformation and fracture behavior of this FCC-metal [29].

Conclusion

Consideration of the findings has shown that iridium single crystals exhibit deformation behavior like usual FCC-metal on the initial/early stages of plastic deformation: clearly visible octahedral slip bands on the back surfaces; high homogeneity of distribution of deformation on the working surface of the samples (no necking); and rectilinear shape of deformation curves. On the other hand, all considerable elongation of the iridium single crystalline samples (up to 70% for $\langle 110\rangle$ axis [16]) happens on these initial/early stages and cleavage is intrinsic fracture mode of iridium single crystals at room temperature. Some of these conclusions/suppositions may be considered as the contradictions with empirical knowledge and, therefore, they are the subjects for further researches including detail TEM study of microstructure of iridium thin foils and its evolution under mechanical stress and heating.

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Appendix

Assuming a shear modulus of approximately 200 GPa for iridium ($c_{11} = 589.9$ GPa $c_{12} = 246.6$ GPa $c_{44} = 266.4$ Gpa—for our samples [30]; $c_{11} = 580$ GPa $c_{12} = 242$ GPa $c_{44} = 256$ Gpa—[31], and calculating a Schmid factor of 0.408 for both the $\langle 100\rangle$ - and $\langle 110\rangle$ orientations (see Fig. 1), the slope of each curve is 0.67 GPa and 0.14 GPa, respectively. The work hardening rates are therefore 3.2×10^{-3} and 6.5×10^{-4} times the shear modulus, respectively, and these values are completely reasonable for an FCC-metal. Hence, according to standard procedure, the strain hardening behavior of iridium seems normal, once it is normalized by the high shear modulus. The Vickers microhardness of iridium single crystals in the initial (undeformed) state is 2500–3000 MPa, whereas this parameter increases up to 4500–5500 MPa after failure of the samples. They are very high magnitudes for a pure FCC-metal, however if these parameters are normalized on shear modulus, the difference disappears. The same may be carried out for $\sigma_{0.2}$ and σ_b, too. These examples confirm the idea that iridium is unusual FCC-metal whose behavior needs in detail study and wide discussion.

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