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On Malleability of Rhenium at Room Temperature

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Abstract. The problem of the malleability of rhenium at room temperature is discussed. It has shown that rhenium manufactured by the mean of the electron beam melting is able to the mechanical treatment by rolling at room temperature. The primary condition for mechanical treatment is the minor share of the tensile stress in the processing scheme. It suppresses the grain boundary sliding in a coarse-grained workpiece of this refractory HCP-metal.

1.Introduction

It is well-known that a hexagonal close-packed (HCP) metal is a ductile material that strengthens weakly under mechanical loading [1,2]. The high mobility of <1120> dislocations on the basal plane and the prismatic plane is the source of the high ductility of HCP-metals [3,4]. The deformation behavior of an HCP-metal depends on the ratio of the lattice parameters c/a [1,2]. Both basal slip and prismatic slip contribute mainly to the plasticity when its c/a is closed to the ideal proportion (1.63). The basal slip is the primary deformation mechanism when its c/a becomes higher than 1.85. Mechanical twinning is another channel for the elastic energy accommodation in an HCP-metal, but its contribution to the total plasticity is minor compared to the dislocation slip [1-4]. Refractory HCP-metal rhenium (T_{meh}=3186°C; c/a = 1,614) does not obey this empirical rule because it is considered an unworkable material at room temperature, at least in the polycrystalline state [5,6]. The cause of this feature of rhenium is still unclear [7] and, hence, needs discussion. This work aims to examine rhenium deformation behavior under rolling and drawing at room temperature because these processing schemes using for the mechanical treatment of metals and alloys.

2.Backgrounds

Rhenium single crystals, which were grown using the zone melting by the electron beam, demonstrate considerable plasticity under tension due to the basal slip and the prismatic slip, while twins with different orientations are observed in the samples [8,9]. Simultaneously, polycrystalline rhenium cannot be processed at room temperature due to its low plasticity. The cause for such behavior may be the high work hardening of rhenium under loading [6]. Is it the intrinsic property of rhenium, or is it the problem connected with the rhenium samples' manufacturing technology? The answer is troubled because obtaining the polycrystalline rhenium samples from the ingot manufacturing by the electron-beam melting (EBM) through mechanical treatment is practically impossible. Therefore, the data on polycrystalline rhenium mechanical properties were obtained on the specimens produced using the powder metallurgy (PM) [5]. Nevertheless, at 1200°C polycrystalline rhenium behaves like a usual HCP-metal at elevated temperatures [10].

Transmission electron microscopic (TEM) study of polycrystalline rhenium has shown that the dislocation slip is the primary contributor to the plasticity under compression and tension at room temperature [11,12]. On the other hand, the interaction between grain boundaries and twins may be

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Journal of Physics: Conference Series

considered as the cause of the high work hardening in rhenium and, perhaps, its low malleability [13]. However, according to the empirical theory, an HCP-metal must be ductile, and it cannot demonstrate a high work-hardening [1-4].

The recent examination of the mechanical properties of polycrystalline rhenium at room temperature has shown that its deformation behavior depends on the manufacturing technology and the share of tensile stress in the loading scheme [14]. Under bending, coarse-grained EBM rhenium exhibits brittle deformation behavior, while PM rhenium behaves like a ductile metal [15]. The cause of the low plasticity of EBM rhenium is the grain boundary sliding (GBS), which is the intrinsic property of a coarse-grained HCP-metal. Both EBM rhenium and PM rhenium demonstrate ductile behavior under shear testing, despite the failure due to GBS [16]. No considerable work-hardening occurred in the polycrystalline rhenium samples under bending and shearing. EBM rhenium withstands severe deformation accompanied by considerable work-hardening under the high-pressure torsion (HPT), while PM rhenium behaves like a brittle material under these conditions [14]. It means that polycrystalline rhenium possesses considerable plasticity resource, which could be suppressed due to such factors as manufacturing technology, the portion of tensile stress in the loading scheme, and GBS coarse-grained samples.

3.Experimental

The rolling and the drawing, which combine compressive and tensile stresses, are mechanical processing technology parts. Therefore, they were chosen as the deformation schemes for polycrystalline rhenium at room temperature.

Refractory metal deformation behavior is highly sensitive to a tiny quantity of non-metallic contaminants in a metallic matrix. Such dangerous impurities as carbon, hydrogen, and oxygen can induce the dramatic lowering of the grain boundary cohesive strength and, hence, the drop of strength and plasticity of a metallic material [17]. This circumstance makes the processing of refractory metals more complicated than metals with low and average melting points. Therefore, the stage of deep refining is the most critical procedure in the manufacture of rhenium and other refractory metals [18]. EBM is an effective method for the pyrometallurgical refining of refractory metals from non-metallic impurities. EBM ingots with a coarse grain structure can be used as workpieces to manufacture rolling and wire from such refractory metals is PM [5]. Therefore, both high pure PM rhenium and EBM rhenium were used as the model materials in this work. The content of non-metallic impurities in the EBM rhenium samples was about 10ppm, which allowed excluding the grain boundary brittleness from possible causes of a low malleability of rhenium.

Rhenium samples were cut using the spark erosion apparatus either from EBM ingot (grain size of $5\div10$ mm) or from PM rod (grain size of $10 \ \mu$ m). EBM workpieces for the rolling had the ingot's shape and a thickness of 0.5mm (figure 1, initial). The drawing samples had a parallelepiped shape with a size of $50x3x3 \ mm^3$ (figure 2a). Mechanical processing of rhenium was carried out at room temperature by UralInTech (Yekaterinburg, Russia) on iridium technological regimes. The surfaces of samples, including fracture surfaces, were documented with the help of a metallographic microscope and a scanning electron microscope (JSM 6390LV) before and after testing.

Vickers microhardness of the samples was measured in the initial state and after loading for every sample. Vickers microhardness of EBM rhenium ingot was about 6GPa, while after the spark erosion cutting, it reached 7GPa. Vickers microhardness of PM rhenium was similar to EBM metal. Therefore, it may be accepted that the value of 7GPa is Vickers microhardness of rhenium in the undeformed state.

4.Results

The samples prepared from PM rhenium were separated into many pieces under rolling and drawing at room temperature practically without preliminary deformation. It means that these schemes cannot be used for the mechanical treatment of PM rhenium at room temperature. Indeed, the working temperature for the processing of PM rhenium is about 700-1000°C.

EBM rhenium workpieces withstood either a few passes of $3\div5\%$ per each or the sole pass of $15\div20\%$ of the cold rolling without the failure (figure 1, rolling I). Their Vickers microhardness increased up to $7,5\div8,5$ GPa under the processing. Further rolling was stopped because it led to the damage of the rolling mill's rolls. 30 minutes annealing of the workpieces in vacuum at 1400°C allows repeating the cold

rolling procedure up to the deformation of 40-70%. The samples began bending under the processing stage (figure 1, rolling II), while their microhardness reached 10MPa. The cracks, whose growth could induce the workpiece's failure, advanced in the grain boundaries (GBs). It should be noted that thin workpieces of such ductile metals like zinc and led were prone to similar behavior under cold rolling.



Figure 1. EBM rhenium workpiece under the cold rolling: (i) initial state; (ii) rolling on 18%; (iii) rolling on 45% (after annealing 30mnutes at 1400°C).



Figure 2. EBM rhenium workpiece under the cold drawing: (a) initial state; (b) drawing.

The images of the EBM workpiece after the drawing are given in figure 2b. After processing, the workpieces' edges became rolled, while it had a rectangular section in the initial state (figure 2a). Besides, the drawing induced the bending of the workpieces. The value of the micrihardness of the workpieces did not change under processing. Metallographic analysis has shown that cracks began growing in GBs in the bending areas (figure 3a). SEM examination of the fracture surfaces allows concluding that the fracture mode of EBM samples under the drawing is the intergranular fracture, where GBs contained many deformation tracks (figure 3b). However, this cracking cannot be estimated as a catastrophic or fast process because the samples with such cracks never separated on many small pieces under the drawing as it happened with PM samples.

049 doi:10.1088/1742-6596/1945/1/012049





Figure 3. EBM rhenium workpiece under the cold drawing: (a) back surface; (b) fracture surface.

5.Discussion

Obtained findings have shown that EBM rhenium exhibits an ability to withstand considerable plastic deformation under cold rolling despite its inclination to GBS. The shape of the workpieces is an essential factor, which allows suppressing this negative effect under rolling. Indeed, the plane stress state takes place in this case, when the GB cracking brakes [20]. On the contrary, the samples' geometry for the drawing is friendly to the craking in the GBs. However, GB cracking is not induced by non-metallic impurities, while its cause is GBS. This EBM rhenium feature confirms that rhenium's plasticity depends on the share of tensile stress in the loading scheme [14-16]. Also, no arguments were found that the cause of the low malleability of rhenium is its work-hardening.

Concerning the malleability of rhenium, it may be stated that EBM metal is able to the mechanical treatment at room temperature. The primary condition for the malleability of coarse-grained rhenium is the minor share of tensile stress in a deformation scheme because it allows suppressing the cracking in

Journal of Physics: Conference Series

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the processing scheme. Also, the cold rolling should be combined with the short term annealing of deformation defects. This property of EBM rhenium opens an opportunity to obtain the fine-grained structure in the ingots using the combination of the forging and the recrystallized annealing, as it takes place in the case of iridium [21]. It is the prospective way the solving the problem of the processing of rhenium because GBS does not happen in the fine-grained samples of an HCP-metal.

PM metal, which was used in this work, cannot be processed at room temperature. However, it does not mean that PM technology does not apply to rhenium. First, PM rhenium exhibits ductile deformation behavior under bending and shearing at room temperature. Second, it seems that many different regimes of the workpiece preparation, including their mechanical properties, should be examined in detail before such a conclusion will be done. Third, industrial experience has shown that PM rhenium is successfully processed at elevated temperatures.

6.Conclusion

EBM rhenium can take the mechanical treatment by rolling at room temperature. The principal condition for treatment is the minor share of the tensile stress in the processing scheme because it suppresses GBS in a coarse-grained HCP-metal.

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