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Microstructure evolution and mechanical properties of the compressively deformed AgInCd alloy during annealing treatment

Weicai Wan[a], Hongsheng Chen[c,*], Haibo Wang[b], Chongsheng Long[c], Qingzhu Sun[b], Yuhe Li[a], Guangzhao Li[a], Jiandong Luo[c]

[a] School of Materials Science and Engineering, Xihua University, Chengdu 610039, PR China
[b] Panzhihua International School of Vanadium and Titanium, Panzhihua College, Panzhihua 617000, PR China
[c] Science and Technology on Reactor Fuel and Materials Laboratory, Nuclear Power Institute of China, Chengdu 610213, PR China

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ABSTRACT

The microstructure evolution and mechanical properties of the compressively deformed AgInCd alloy during annealing treatment have been investigated in the present study. In the deformed AgInCd alloy, many deformation markings are formed and these deformation markings are mainly composed of nanoscale deformation twins. The AgInCd alloy exhibits a significant work hardening capability because of the formation of deformation twins. The recrystallization temperature of the deformed AgInCd alloy is between 300 °C and 350 °C. In the AgInCd alloy annealed at 300 °C, the deformation twins are eliminated and a large number of dark strips consisting of stacking faults are presented in the un-recrystallized regions, indicating that the deformation twins are transformed into stacking faults by the detwinning mechanism. The annealing twins show higher thermal stability than the deformation twins. The strength and microhardness of the deformed AgInCd alloy gradually decrease with the increase of annealing temperature.

1. Introduction

AgInCd alloy with compositions of 80 wt% Ag, 15 wt% In, and 5 wt % Cd is widely used as the control rod material in the pressurized water reactors, because this alloy has superior nuclear properties, appropriate physical and mechanical properties and good irradiation resistance in comparison to other neutron absorber materials such as boron carbides, boron-stainless steels and hafnium alloys [1–5]. A large number of irradiation data and operating experiences have been accumulated for the AgInCd control rods in the commercial pressurized water reactors since this alloy was firstly developed for power reactor application in 1950 [6]. Despite of its superior properties, the AgInCd control rods were blocked by the guide tubes several times in the long service history [3]. Many factors might cause the block phenomenon of AgInCd control rods, such as the irradiation-induced swelling, plastic deformation (including creep) and so on. Previous studies indicated that the effective radial swelling value of control rods is larger than the predicted swelling value (irradiation-induced swelling) [7]. Thus plastic deformation may play an important role on the block of AgInCd control rods. During the operation process of plant, compressive stresses are exerted on the AgInCd control rods as a result of themselves gravity and the pressure springs of control rod ends. It is generally believed that plastic deformation is expected to occur in the AgInCd control rods under the compressive stresses [3,8,9]. Plastic deformation can give rise to the larger diameter at the lower end of the fuel rods, and which may result in the 'block' phenomenon. Therefore, it is significant for the nuclear reactor safety to understand the behaviors of the AgInCd alloy in the in-pile environment.

Recently, the compressive deformation mechanisms of the AgInCd alloy at room-temperature were explored preliminarily and it was found that the dominant deformation mechanisms transformed from the dislocation slipping and multiplication into deformation twinning in the deformed AgInCd alloy when the compressive strain reached 50% [8]. The formation of nano-twins has a significant impact on the properties and related behaviors of the AgInCd alloy. On the other hand, the service temperatures of the AgInCd control rods usually range between 300 °C and 350 °C in commercial reactors, which are higher than their recrystallization temperatures (~275 °C) [6,10]. Recovery and recrystallization can occur inevitably for the deformed AgInCd alloy at these temperatures. Hence, more careful attentions should be given to the recovery and recrystallization behaviors of this alloy.

Some studies have been conducted to deal with the recrystallization behaviors of Ag-based alloys. In early stage, Bailey and Hirsch [11] found that small difference in the impurity content could result in the

* Corresponding author.
E-mail address: hschen@npic.ac.cn (H. Chen).

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different recrystallization temperatures of the deformed polycrystalline silver. Haessner et al. [12] believed that some solute atoms (Al, Cu, Sb, Sn) with distribution coefficients $k < 1$ reduced the stored energy and increased the recrystallization temperature of the Ag-based alloys, and whereas other solute atoms (Au, Mn, Pd) with distribution coefficients $k > 1$ would cause the opposite effect (Distribution coefficient is the ratio of concentration of a solute in two phases (usually liquid and solid phases) at equilibrium). Recent experiment also revealed that one of solute atoms with distribution coefficients $k < 1$, Cu, decreased the recrystallization temperature of Ag-based alloys, in which recrystallization started at annealing temperature as low as 250 °C in the copper–silver bimetallic system [13]. It is well known that the AgInCd alloy has face-centred cubic (*fcc*) structure with very low stacking fault energy (< 22 ergs/cm^2 [8]). The recrystallization mechanisms of the deformed *fcc* metals with low stacking fault energy are usually difficult to characterize, because the formation of shear bands and fine deformation twinning are usually promoted, which can bring about the complexity of the deformation microstructures in the deformed metals [14,15]. In the deformed AgInCd alloy, a large number of staking faults and deformation twins were also observed in the previous study [8]. At present, to the authors' knowledge, the recovery and recrystallization behaviors of the deformed AgInCd alloy have not yet been clear. Therefore, the present paper investigates the recovery and recrystallization behaviors of the AgInCd alloy with compressive deformation stain, and mainly focuses on the microstructure evolution and mechanical properties of the compressively deformed AgInCd alloy during annealing treatment.

2. Experimental Procedure

AgInCd alloy with the composition Ag–15 wt% In–5 wt% Cd was cast firstly from the purity components (99.95 wt% Ag, 99.99 wt% In and 99.99 wt% Cd) by sealed vacuum melting. Then the casting ingots were machined into the cylindrical specimens with size of Φ 8.6 mm × 10 mm. Compressive deformation tests of the AgInCd alloy with recrystallized structure were performed at room temperature by using a universal tensile testing machine equipping with a domestic compressive experimental fixture, this fixture was introduced in Ref. [8]. During the compressive deformation testing, the cross-head displacement rate was constant with an initial strain rate of $10^{-1}\,s^{-1}$. All the cylindrical specimens were compressively deformed with an engineering strain of 50% in the axial direction. After the compressive deformation testing, the deformed AgInCd specimens were annealed in a vacuum furnace at moderate temperatures ranging from 300 °C to 450 °C with a holding time of 1 h. These AgInCd samples were heated and cooled under a rate of ~10 °C min^{-1}. During the whole annealing process, the vacuum pressure was always $< 1 \times 10^{-3}$ Pa.

After the annealing treatment, the microhardness of the deformed and annealed AgInCd specimens was measured by a UHL HVS-50 Vickers tester, a 200 g load was exerted on the polished surface of the specimens for duration of 10 s during the microhardness testing. Ten microhardness tests were performed for each specimen. In view of the smaller size of the cylindrical specimens, the mini-sized tensile specimens were machined by the electrical discharge wire cutting to evaluate the tensile properties of the undeformed, deformed and annealed AgInCd alloys. The geometry and dimensions of the mini-sized tensile specimens are shown in Fig. 1. The tensile experiments were conducted at room temperature with a stain rate of $10^{-4}\,s^{-1}$ by using a universal tensile testing machine. To check the reproducibility of the tensile results, three tensile experiments were performed for the same specimen. For the microstructural observation, these AgInCd specimens with different conditions were firstly metallographically polished by standard polishing techniques and then etched by an etching solution (H_2SO_4: 10 ml; saturated aqueous solution of $K_2Cr_2O_7$: 100 ml; saturated aqueous solution of NaCl: 2 ml). An Olympus OLS4000 three-dimensional laser scanning microscope and a FEI NOVA NanoSEM400 scanning electronic microscope (SEM) were used to examine the microstructure of the etched specimens. Based on the microstructural images, a dimensional measurement software (Mias, developed by Sichuan University) was used to measure the grain size. In order to obtain good statistics, > 300 grains are measured for each specimen. Because many twins exist in the AgInCd alloy and these twin boundaries can affect the measurement of grain size, we manually excluded these twin boundaries according to their straight characteristics during the process of grain size measurement. A JOEL JEM-2100 transmission electron microscope (TEM) was also used to characterize the detailed microstructure of the AgInCd specimens at an acceleration voltage of 200 kV. The phase structures of the AgInCd specimens were analyzed by the selected area electron diffractions, which were taken from these regions indicated by the white circles in corresponding TEM images. To obtain the foils for TEM observation, the specimens were cut and ground to approximately 100 μm thickness by the silicon carbide paper, and then thinned by means of double-jet electrolytic polishing.

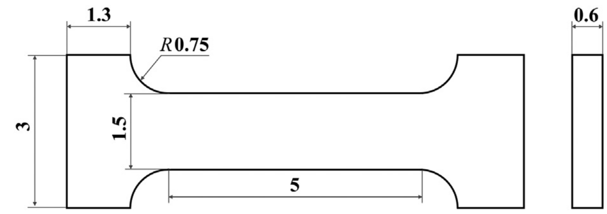

Fig. 1. Geometry and dimensions of mini-sized tensile specimen (in mm).

3. Results and Discussion

3.1. Microstructure and Mechanical Properties of the Undeformed and Deformed AgInCd Alloys

Fig. 2 shows the typical microstructure of the undeformed AgInCd alloy. It can be seen that an embossed morphology is presented in the SEM micrograph, as shown in Fig. 2a. This is because that the different grains possess different crystallographic orientations, it is believed that the corrosion rate has strong dependence on the crystallographic orientations for many metals and alloys, and the close-packed planes usually exhibit more corrosion resistant than other crystallographic planes [16,17]. By the way, the twin boundaries can be distinctly displayed in the etched surface resulting from the different crystallographic orientations between the matrix and twin in the same grain. These annealing twins are easily formed in the preparation process of AgInCd alloy because this alloy has an extremely low stacking fault energy (< 22 ergs/cm^2) [8]. The TEM micrograph shown in Fig. 2b reveals that a small number of dislocations are retained in the vicinity of twin boundaries. When a lattice dislocation encounters the twin boundary, local dissociation reactions can occur and twinning dislocations can be created in these processes [18,19]. The twinning dislocations possibly belong to the sessile residual dislocation, for example, the *Frank* partial dislocation for *fcc* metals [18]. Therefore, some dislocations are existed near the twin boundaries in the undeformed AgInCd alloy.

The microstructure of the severely deformed AgInCd alloy is shown in Fig. 3. The optical micrograph and SEM micrograph show that the grains are difficult to identify and the annealing twin boundaries become bent and blurry in the deformed alloy. This is because that during the deformation process, successive gliding dislocations interact with the grain boundaries and twin boundaries, and these interactions can result in the translation and curve of the grain boundaries and twin boundaries [20,21]. Many deformation markings also can be observed in the deformed grains, as shown in Fig. 3b. The deformation markings are often detected in the deformed *fcc* alloys, and usually consist of packets of the slip bands and deformation twins [22]. TEM micrograph