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ARTICLE

Effects of Low-Temperature Aging on Dimensional Stability of Beryllium

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Abstract: To improve its dimensional stability, the microstructure and residual stress of hot isostatically-pressed beryllium were regulated by low-temperature aging. Results show that after low-temperature aging, the dimensional shrinkages of hot isostatically-pressed beryllium, annealed beryllium, and thermal-cold cycled beryllium are evidently reduced. Various stabilizing mechanisms, including dislocation homogenization and coordinated grain micro-deformation, are observed during low-temperature aging, which effectively improve the dimensional stability of hot isostatically-pressed beryllium.

Key words: beryllium; low-temperature aging; microstructure; dimensional stability

Due to its characteristics of low density, high elastic modulus, high specific strength, beryllium has become a key material for inertial navigation instruments[1–3]. There is a high requirement for the dimensional stability of beryllium, because a slight dimensional change will seriously damage the accuracy and stability of inertial navigation[4–5]. Therefore, it is eager to explore an effective method to improve the dimensional stability of beryllium. Low-temperature aging is a common heat treatment used to promote strength and hardness and to reduce residual stress[6–8]. The microstructure and residual stress change slowly during low-temperature aging[9]. Therefore, low-temperature aging is a possible method to improve the dimensional stability of hot isostatically-pressed beryllium.

There is few report on the effect of low-temperature aging on the microstructure and residual stress of beryllium. The questions of whether and to what extent the low-temperature aging can improve the dimensional stability of hot isostatically-pressed beryllium, and what the dimensional stabilizing mechanisms are were studied to provide a theoretical basis for improving navigation accuracy and stability.

1 Experiment

Hot isostatically-pressed beryllium was used as the raw material, and the chemical composition conformed to the requirements of GJB 1539A for grade RJY-55. Hot isostatically-pressed beryllium was untreated after isostatic pressing and referred to as HIP beryllium[10]. Annealed beryllium was obtained by annealing the HIP beryllium at 800 °C for 2 h. The thermal-cold cycled beryllium was obtained by cycling for 6 times at –60 (1 h) –150 °C (1 h), which was referred to as TCC beryllium.

Low-temperature aging and dimensional changes of thermal cycles were carried out on the thermal expansion analyzer. The temperatures of low-temperature aging were 100, 140, 180, 220, 260 and 300 °C. The temperature range of thermal cycles was 25–150 °C, and the heating and cooling rates were less than 5 °C/min. The residual stress, misorientation, dislocation density, and dislocation morphology of beryllium were analyzed by Raman spectrometer, nano indentation, electron backscatter diffraction (EBSD), and transmission electron microscope (TEM)[11–13].

2 Results and Discussion

2.1 Dimensional changes during low-temperature aging and thermal cycles of berylliums

Fig. 1 shows the dimensional changes during different treatments of beryllium. Fig. 1a shows the dimensional

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changes of HIP beryllium, annealed beryllium, and TCC beryllium during aging in the range of 100–300 °C. A similar trend of total dimensional changes during aging of HIP beryllium, annealed beryllium, and TCC beryllium can be observed. With the continuous increase in temperature, the dimensional changes increase and then decrease. The dimensional change of HIP beryllium aged at 180 °C is -46×10^{-5}, and the dimensional changes of HIP beryllium aged at 100 and 300 °C are -16×10^{-5}, -9×10^{-5}, respectively. Compared with HIP beryllium, the dimensional changes during aging of annealed beryllium and TCC beryllium are reduced by 1%–5%, 5%–20%, respectively.

Fig. 1b shows the dimensional changes during the thermal cycles of HIP beryllium, annealed beryllium, and TCC beryllium. The heating and cooling rates of annealing treatment are fast due to high annealing temperature, which will promote thermal mismatch stress between beryllium oxide and beryllium matrix [5]. The temperature range of the thermal-cold cycling is small, which is not enough to introduce a large external stress superposition to eliminate the original stress state. Therefore, before low-temperature aging, dimensional changes of annealed beryllium and TCC beryllium are slightly smaller than that of HIP beryllium. Low-temperature aging has a significant dimension stabilizing effect. After low-temperature aging at 300 °C, the dimensional changes of HIP beryllium, annealed beryllium, and TCC beryllium are significantly reduced. The cumulative dimensional changes during 10 thermal cycles of HIP beryllium, annealed beryllium, and TCC beryllium are reduced to less than 18×10^{-6}, indicating a stable state. After low-temperature aging in the range of 100–300 °C, the dimensional changes during thermal cycles of HIP beryllium are significantly decreased, and show a law of decrease with the increase in aging temperature.

2.2 Residual stress of HIP beryllium after low-temperature aging

Fig. 2 shows the residual stress of HIP beryllium after different low-temperature aging. As shown in Fig. 2a, the Raman shift of the stress-free beryllium is 455 cm^{-1}, and that of HIP beryllium is 459.6 cm^{-1}, which indicates the existence of residual compressive stress caused by deformation during the isostatic pressing process [11]. After low-temperature aging in the range of 100–300 °C, all the Raman shift of HIP beryllium remains around 459 cm^{-1}, indicating that the residual stress does not decrease. As shown in Fig. 2b, compared with the stress relieved annealed beryllium as the stress-free sample, the residual compressive stress of HIP beryllium is 120 MPa. After aging in the range of 100–300 °C, the residual stress of HIP beryllium does not decrease significantly. Therefore, low-temperature aging cannot effectively eliminate the residual stress of HIP beryllium. Therefore, the dimensional changes during low-temperature aging were mainly caused by the change of microstructure.

2.3 Misorientation and dislocation of HIP beryllium after different low-temperature aging

Fig. 3 shows the misorientations and dislocation density of HIP beryllium after different low-temperature aging. The misorientations of HIP beryllium are multiplex, including 50% of substructured grains, 24% of deformed grains and

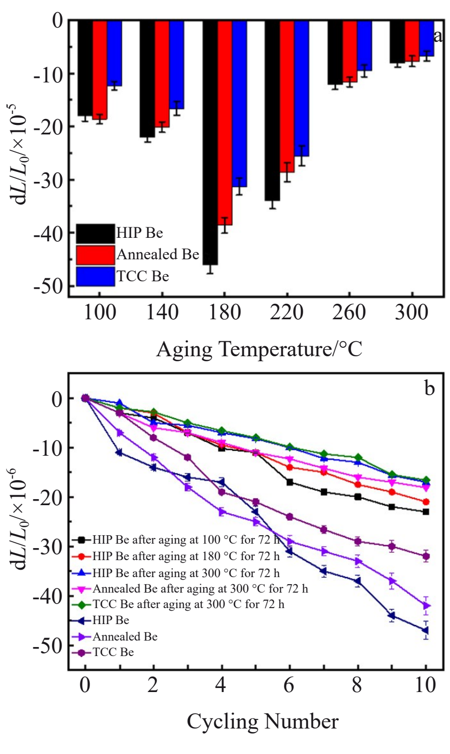

Fig.1 Dimensional changes of beryllium during aging in the range of 100–300 °C (a) and thermal cycles (b)

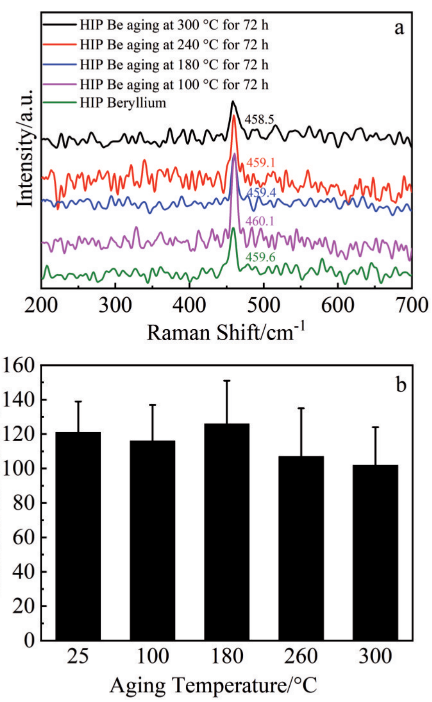

Fig.2 Residual stress of HIP beryllium after different low-temperature aging: (a) Raman method and (b) nano indentation method

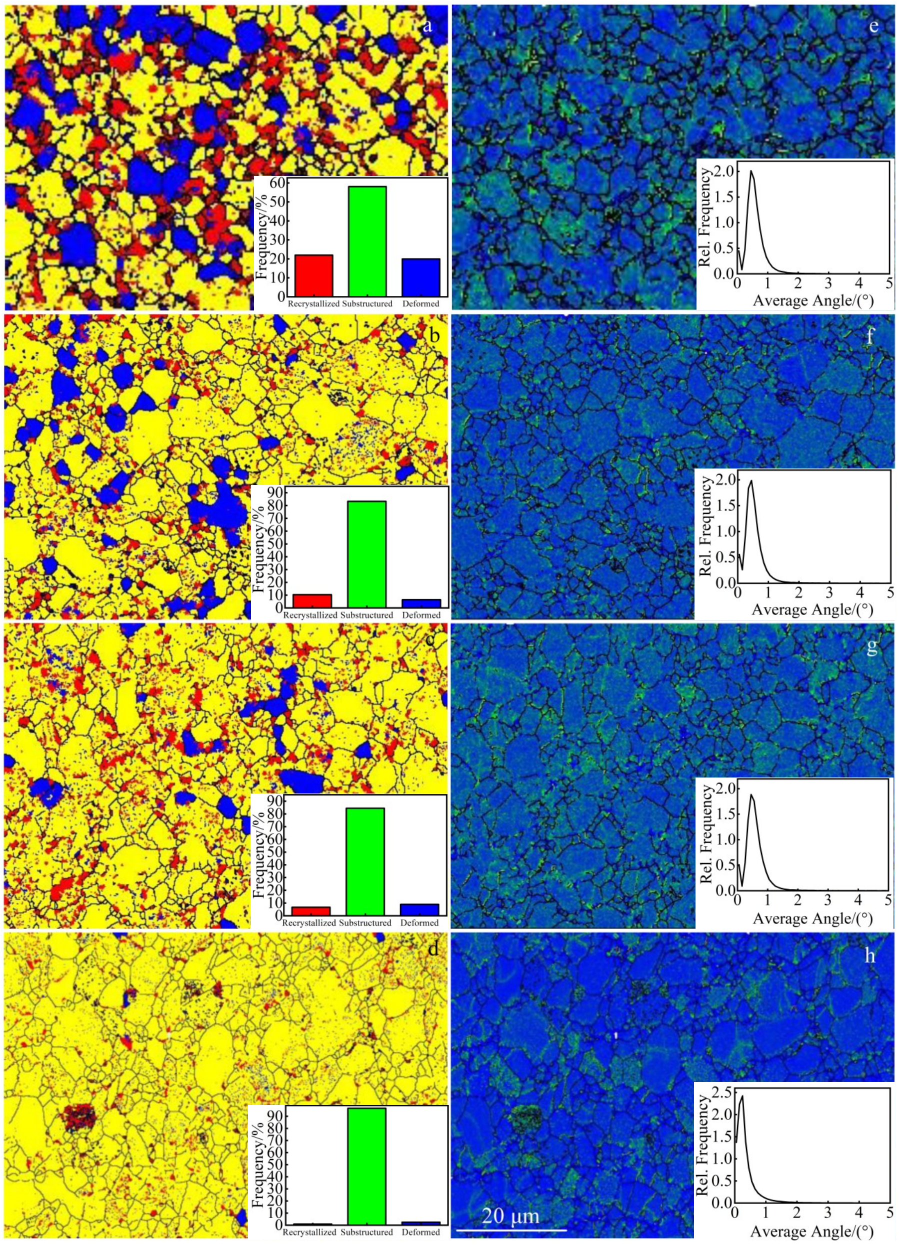

Fig.3 Misorientations (a–d) and dislocation density (e–h) of HIP beryllium: (a, e) before aging, (b, f) aging at 100 °C, (c, g) aging at 180 °C, and (d, h) aging at 300 °C

25% of recrystallized grains, as shown in Fig. 3a. After aging in the range of 100 – 300 ° C, the misorientations gradually transform into substructure grains. After aging at 300 ° C, the micorientations are all uniform substructure grains, as shown in Fig.3b–3d. The changes of misorientations after low-temperature aging are caused by the coordinated micro-deformation, which leads to the dimensional shrinkage of beryllium. After aging at 300 ° C, the misorientations are even, which makes it difficult to transfer the stress and strain through the grains, increasing the deformation resistance[12].

The dislocation distribution of HIP beryllium is uneven, as shown in Fig. 3e. Since grain boundaries are pinned by BeO and aging temperature is not high enough, the dislocations cannot pass through grain boundary to realize dislocation homogenization between grains. Therefore, after aging in the range of 100 – 300 ° C, the change of dislocation density is small and the distribution uniformity in grains is improved, as shown in Fig.3f–3h.

2.4 Dislocation morphology of HIP beryllium after different low-temperature aging

Fig. 4 shows the TEM images of HIP beryllium after different low-temperature aging. The HIP beryllium is composed of beryllium grains with uneven sizes and nano BeO are distributed discontinuously along the grain boundary. The grain boundaries are relatively straight and the angles of grain boundaries are sharp. There are some dislocation lines in the small-sized grains, as shown in Fig. 4a. The dislocation structure shifts from dispersed line dislocations to entangled

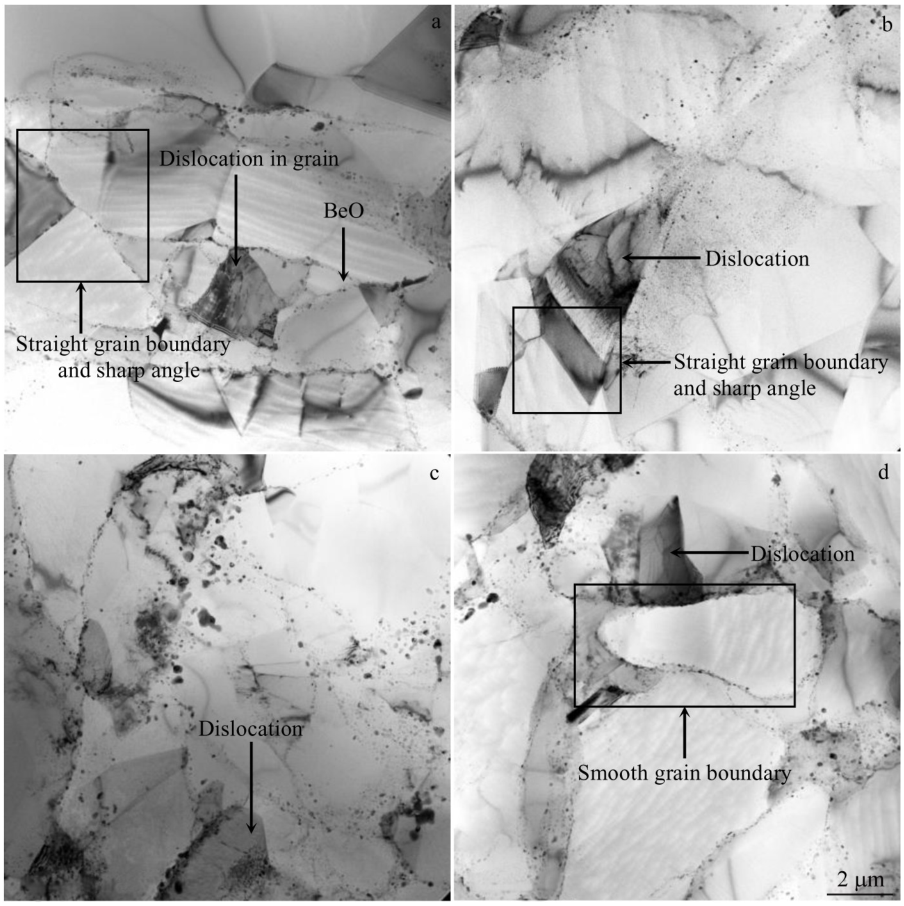

Fig.4 TEM images of HIP beryllium after different low-temperature aging: (a) before aging, (b) aging at 100 °C, (c) aging at 180 °C, and (d) aging at 300 °C

dislocations. Dislocations are bounded by grain boundaries and can only achieve homogenization within the grains. Due to the inability of dislocations to proliferate and annihilate during aging in the range of 100 – 300 °C, the change of dislocation density is small. As the temperature increases up to 300 °C, the grain boundaries become smooth, as shown in Fig. 4b – 4d. The changes of dislocations during low-temperature aging will lead to micro-plastic deformation in grains and a small amount of residual stress relaxation. The dislocations retained after low-temperature aging are relatively stable[13].

3 Conclusions

1) After low-temperature aging, the dimensional changes of HIP beryllium significantly decrease, and show a law of decrease with the increase in temperature.

2) Residual compressive stress exists in HIP beryllium, which will not decrease significantly after low-temperature aging.

3) After low-temperature aging, the misorientations are even, which makes it difficult to transfer the stress and strain through the grains, increasing the deformation resistance. The change of dislocation density is small and the distribution uniformity in grains is improved.

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低温时效对铍材尺寸稳定性的影响

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摘 要：利用拉曼光谱仪、纳米压痕仪、电子背散射衍射电镜以及透射电镜分析低温时效后等静压铍材的残余应力、晶粒取向差、位错密度及位错分布形态，分析低温时效处理对铍材尺寸稳定性的影响。结果表明，低温时效处理后，热等静压铍材、退火铍材和热冷循环铍材在热循环过程中的尺寸收缩均明显减小。低温时效处理过程中铍材尺寸稳定化机制主要是晶粒内位错均匀化及微塑性变形导致取向差均匀化。

关键词：铍材；低温时效；组织结构；尺寸稳定性

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