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LETTER

# Effects of Thermal-Cold Cycling on Dimensional Stability of HIPed Beryllium

**Ren Penghe[1], Xiao Lairong[1,2], Cai Zhenyang[1,2], Tu Xiaoxuan[1], Zhao Xiaojun[1,2]**

*[1] School of Materials Science and Engineering, Central South University, Changsha 410083, China; [2] Key Laboratory of Nonferrous Metal Materials Science and Engineering, Ministry of Education, Changsha 410083, China*

**Abstract:** The dimensional change, residual stress, grain orientation difference, dislocation density, and dislocation distribution of beryllium after different hot isostatic pressing treatments were analyzed by laser length meter, Raman spectrometer, nanoindentation meter, electron backscattered diffractometer, and transmission electron microscope, and the influence of thermal-cold cycling treatment on the dimensional stability of beryllium was analyzed. Results show that the size of the hot isostatic pressed beryllium tends to be stable after 6 cycles of thermal-cold cycling treatment from −100 °C to 150 °C, and it has good dimensional stability. The dimensional stabilization mechanism of beryllium is mainly the homogenization of dislocations within the grain and the homogenization of orientation difference caused by micro-plastic deformation.

**Key words:** beryllium; thermal-cold cycling; microstructure; dimensional stability

Due to its low density, high elastic modulus, and high specific strength, beryllium becomes a key material for the inertial navigation instruments[1–2]. High requirements have been proposed for the dimensional stability of beryllium, because the slight dimensional change may damage the accuracy of inertial navigation[3–5]. Therefore, it is imperative to explore an effective method to improve the dimensional stability of beryllium. Researches present that the dimensional stability is decisively affected by microstructure and residual stress[6–7]. The thermal-cold cycling (TCC) treatment involves the alternate cycle of cold treatment below 0 °C and heat treatment at 100–500 °C, which is considered as an effective way to improve dimensional stability[8–10]. TCC treatment imposes cyclic stress on the material, leading to dislocation movement and phase transformation and improving the dimensional stability[11]. The effects of TCC treatment on the microstructure and residual stress of beryllium are rarely reported. Therefore, the effect induced by TCC treatment on dimensional stability of beryllium after hot isostatic pressing (HIP) was investigated, and the dimensional stabilization mechanisms were also studied in this research.

## 1 Experiment

The commercial HIPed beryllium was used as the raw material, and the composition is listed in Table 1. The beryllium of control group was aged at 300 °C for 30 d, denoted as Control Be. Both Control Be and HIPed beryllium (HIP Be) samples were subjected to different TCC treatments to evaluate the dimensional stability. Three commonly used TCC treatments were selected: heating at 150 °C for 1 h and then cooling at −60, −100, and −196 °C for 1 h, so they were denoted as TCC/150/−60, TCC/150/−100, and TCC/150/−196, respectively. The number of TCC cycles was 0–10[11–12]. After TCC treatments, the dimensional changes during the heat preservation process at 150 °C for 100 h were recorded to evaluate the dimensional stability. Dimensional changes during long-term low-temperature insulation can effectively reflect the dimensional stability after TCC treatments. TCC treatments and heat preservation at 150 °C for 100 h were conducted by a low-temperature thermostat and vacuum drying oven. Dimensional changes, residual stress, microstructure, and dislocation morphologies were characterized by laser length meter, Raman spectrometer, nanoindentation

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Corresponding author: Zhao Xiaojun, Ph. D., Associate Professor, School of Materials Science and Engineering, Central South University, Changsha 410083, P. R. China, Tel: 0086-731-88877499, E-mail: zhaojx@csu.edu.cn

**Table 1 Composition of HIP Be (wt%)**

| Be | Al | Fe | Si | BeO | Other |
|---|---|---|---|---|---|
| 99.1 | 0.02 | 0.06 | 0.02 | 0.9 | Bal. |

meter, electron backscattered diffractometer (EBSD), and transmission electron microscope (TEM)[13].

## 2 Results and Discussion

### 2.1 Dimensional changes of HIP Be during and after TCC treatments

Fig. 1a shows the dimensional changes of Control Be and HIP Be after different TCC treatments, where $L$ is the current length and $L_0$ is the initial length. The dimensional shrinkages of HIP Be samples occur after different TCC treatments. It is reported that the lower the cold treatment temperature of TCC treatment, the slighter the cumulative dimensional changes. It can be seen that the dimension of HIP Be after 6 cycles of TCC/150/−100 treatment is basically stabilized. The dimensions of HIP Be after 10 cycles of TCC/150/− 60 and TCC/150/−196 treatments continue to change. It can be observed that the dimensional changes of Control Be after 10 cycles of TCC/150/−60 and TCC/150/−100 treatments are slight. During TCC/150/−196 treatment, the dimensional change of Control Be is similar to that of HIP Be.

Fig. 1b shows the dimensional changes of beryllium before (initial) and after TCC treatments followed by reheating at 150 °C for 100 h. Initial Control Be and HIP Be were also reheated at 150 °C for 100 h for comparison. The dimensional shrinkage of HIP Be after TCC/150/− 100 treatment followed by reheating is significantly smaller than of HIP Be after TCC/150/−60 and TCC/150/−196 treatments followed by reheating. The dimensional shrinkage of HIP Be after TCC/150/− 100 treatment followed by reheating is only $1.2\times10^{-5}$, which is in a stable state. Compared with that of initial Control Be, the dimensional changes of Control Be after TCC/150/−60 and TCC/150/−196 treatments followed by reheating are slightly smaller, and that of Control Be after TCC/150/−196 treatment followed by reheating is larger. It is clear that the TCC/150/−100 treatment is a suitable dimensional stabilization method for HIP Be processing.

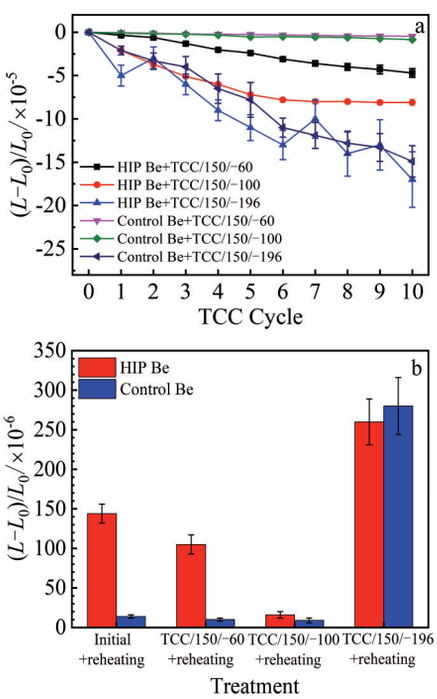


Fig.1 Dimensional changes of different beryllium samples during TCC treatments (a) and before and after TCC treatments followed by reheating at 150 °C for 100 h (b)

### 2.2 Residual stress of HIP Be after different TCC treatments

Fig. 2 shows the residual stress of HIP Be before (initial) and after different TCC treatments. As shown in Fig. 2a, compared with that of the stress-free beryllium (455 $cm^{-1}$)[14], the Raman shift of HIP Be is 459.6 $cm^{-1}$, indicating the existence of residual compressive stress. During TCC treatment, cyclic stress is applied through the impact of temperature cycle to eliminate the original compressive stress and to achieve residual stress relaxation. After TCC/150/− 60 and TCC/150/− 196 treatments, the Raman shifts of HIP Be move to 459 and 460.5 $cm^{-1}$, respectively. The Raman shift change of HIP Be is small, which indicates that the external stress provided by TCC cannot effectively eliminate the original residual stress. After TCC/150/−100 treatment, the Raman shift of HIP Be moves to 456.8 $cm^{-1}$, suggesting that the stress provided by the cold and thermal cycles is appropriate and thereby achieving the stress removal effect.

As shown in Fig. 2b, the residual compressive stress of initial HIP Be is 120 MPa. After TCC/150/−100 treatment, the

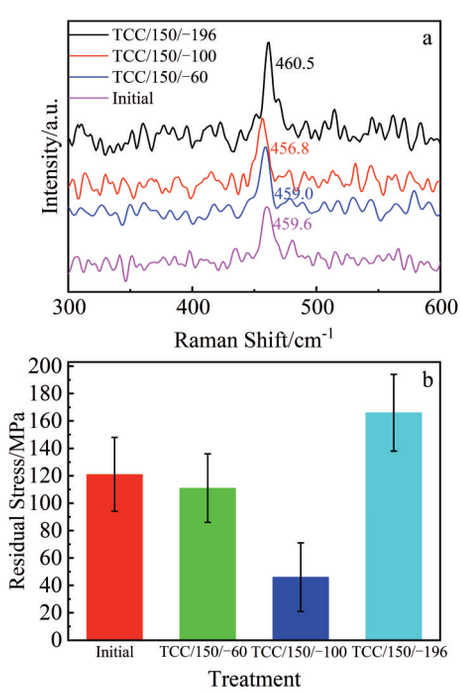


Fig.2 Raman spectra (a) and residual stress (b) of HIP Be samples after different TCC treatments

residual stress of HIP Be decreases significantly. Therefore, TCC/150/−100 treatment can effectively eliminate the residual stress of HIP Be.

### 2.3 Misorientation and dislocation of HIP Be after different TCC treatments

Fig. 3a – 3d show the misorientations of HIP Be after different TCC treatments. The misorientations of HIP Be are multiplex, including 58% substructured grains, 22% deformed grains, and 20% recrystallized grains, as shown in Fig.3a and 3e. The deformed grains almost disappear after different TCC treatments. Recrystallized grains are still retained in HIP Be after TCC/150/−60 and TCC/150/−196 treatments, as shown in Fig.3b and 3d, respectively. The misorientations of HIP Be after TCC/150/−100 treatment are basically composed of substructured grains, as shown in Fig.3c. The changes in grain misorientations after TCC treatments are caused by coordinated micro-plastic deformation and grain rotation, which leads to dimensional shrinkage. After TCC/150/−100 treatment, the misorientations are homogeneous. Therefore, the stress and strain become difficult to transfer between grains, resulting in the improvement of dimensional stability.

The dislocation densities of HIP Be are shown in Fig.4a–4d. It can be seen that the dislocation density of initial HIP Be is inhomogeneous, as shown in Fig. 4a. After TCC/150/−60 treatment, the change in dislocation density is small and the distribution uniformity improves, as shown in Fig. 4b. Compared to that of the initial HIP Be, lower dislocation density can be observed after TCC/150/−100 treatment, as shown in Fig. 4c. The dislocation density slightly increases after TCC/150/−196 treatment, as shown in Fig. 4d. The changes in dislocations during TCC treatment may lead to micro-plastic deformation and residual stress relaxation.

### 2.4 Dislocation morphology of HIP Be after different TCC treatments

Fig.5 shows TEM morphologies of HIP Be before and after different TCC treatments. The HIP Be is composed of

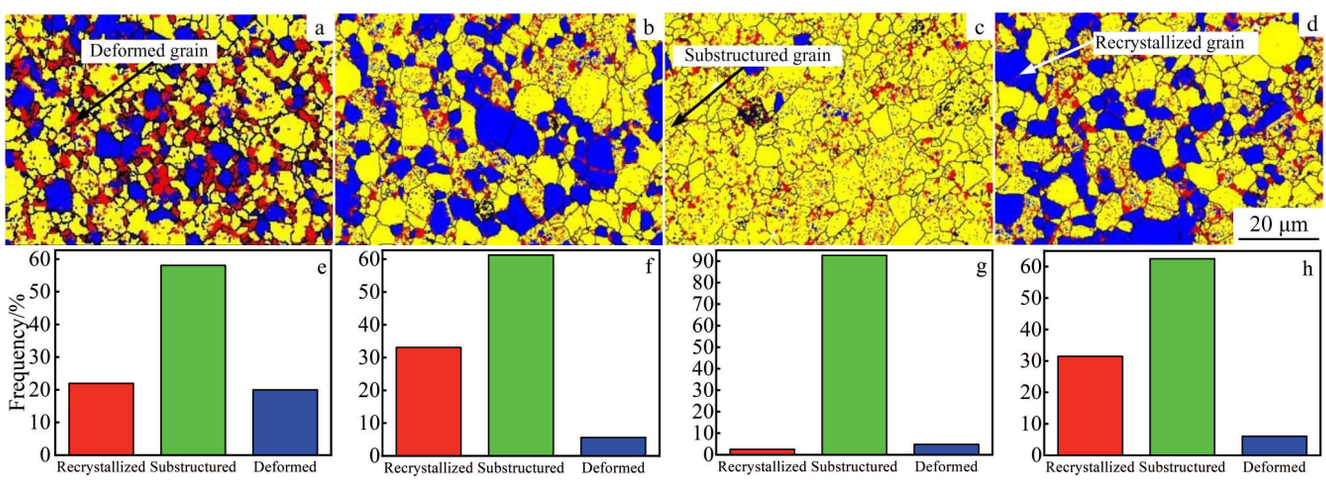


Fig.3 Misorientations (a–d) and misorientation distributions (e–h) of HIP Be before (a, e) and after TCC/150/−60 treatment (b, f), TCC/150/−100 treatment (c, g), and TCC/150/−196 treatment (d, h)

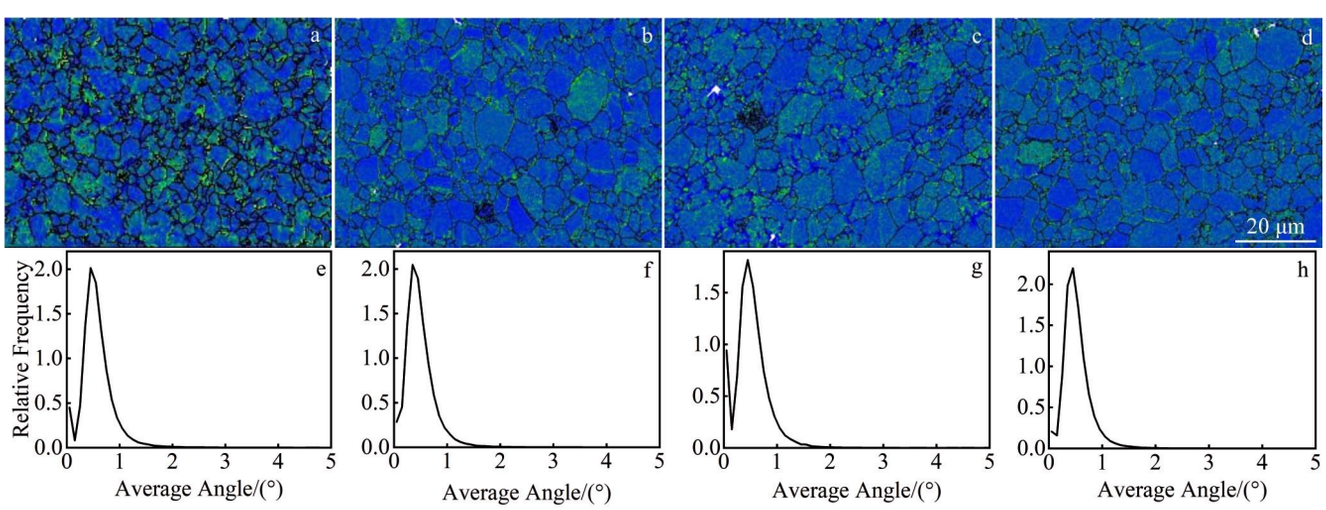


Fig.4 Dislocation densities (a–d) and average angle distributions (e–h) of HIP Be before (a, e) and after TCC/150/−60 treatment (b, f), TCC/150/−100 treatment (c, g), and TCC/150/−196 treatment (d, h)

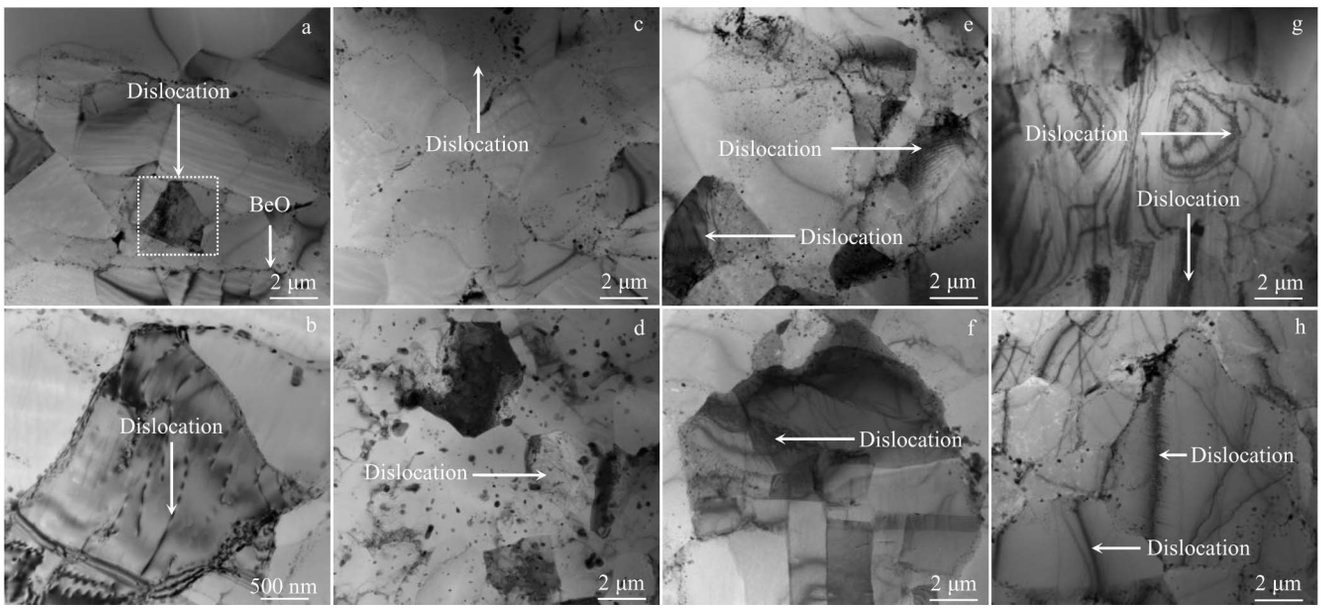


Fig.5 TEM morphologies of HIP Be before (a–b) and after cold cycling (c, e, g) and thermal cycling (d, f, h) of TCC/150/−60 treatment (c–d), TCC/150/−100 treatment (e–f), and TCC/150/−196 treatment (g–h)

beryllium grains with uneven sizes and nano-BeO distributed discontinuously along the grain boundary. The grain boundaries are relatively straight and the angles of grain boundaries are small, as shown in Fig. 5a. There are some dislocation lines in the small-sized grains[15], as shown in Fig.5b. The changes in dislocations of HIP Be after TCC/150/−60 treatment are not obvious, as shown in Fig.5c–5d. During TCC/150/−100 treatment, it can be observed that the number of dislocations in HIP Be increases after cold cycling and decreases after thermal cycling. The grain boundaries also become smooth, as shown in Fig. 5e – 5f. The dislocation entanglement of HIP Be after cold cycling at −196 °C is obvious, so as the remaining dislocations after thermal cycling at 150 °C, as shown in Fig. 5g and 5h, respectively. In TCC treatment, the dislocations are generated during the cold cycling, and then they are decreased during the thermal cycling, resulting in the continuous annihilation of the original dislocations in HIP Be. After several cycles, a stable dislocation state with minimum micro-stress and macro-stress is generated. It is clear that the dislocations can be effectively regulated by TCC/150/−100 treatment.

## 3 Conclusions

1) The dimensional shrinkages are increased with the decrease in the cold cycling temperature of TCC treatment. The dimensional shrinkage of HIP Be after TCC/150/−100 treatment followed by reheating is only $1.2\times10^{-5}$, which is in a stable state. Therefore, TCC/150/−100 treatment is a suitable dimensional stabilization method for HIP Be.

2) Residual compressive stress exists in HIP Be, which decreases significantly after TCC/150/−100 treatment.

3) After TCC treatment, the misorientations are homogeneous and the distribution uniformity improves, which makes it difficult to transfer the stress and strain through the grains, therefore increasing the deformation resistance.

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# 冷热循环处理对热等静压铍材尺寸稳定性的影响

任鹏禾[1]，肖来荣[1,2]，蔡圳阳[1,2]，涂晓萱[1]，赵小军[1,2]

(1. 中南大学 材料科学与工程学院，湖南 长沙 410083)

(2. 有色金属材料科学与工程教育部重点实验室，湖南 长沙 410083)

**摘　要：**利用激光测长仪、拉曼光谱仪、纳米压痕仪、背散射衍射电镜以及透射电镜分析不同处理状态等静压铍材的尺寸变化、残余应力、晶粒取向差、位错密度及位错分布形态，分析冷热循环处理对铍材尺寸稳定性的影响。结果表明，热等静压铍材在−100~150 ℃冷热循环6次后尺寸趋于稳定，具有良好的尺寸稳定性。冷热循环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；冷热循环处理；组织结构；尺寸稳定性

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作者简介：任鹏禾，男，1993年生，博士生，中南大学材料科学与工程学院，湖南 长沙 410083，电话：0731-88877499，E-mail：csuherb@csu.edu.cn