

# Effect of aging temperature on dimensional instability of invar alloys

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In this article, the effect of aging process on dimensional instability of Invar alloys was analyzed. When the aging temperature ranged from 200°C to 500°C, the organization of the Invar alloy was austenite phase  $\gamma$ -Fe. The residual stress of the Invar alloy also decreased significantly with the increase of the aging temperature. The coefficients of thermal expansion (CTE) of the Invar alloy did not change obviously with the increase of the aging temperature when the aging temperature was below 400°C. However, the CTE of the Invar alloy increased significantly with the increase of the aging temperature when the aging temperature was above 400°C. After aging at 500°C, the average CTE of the alloys increased 100% and the dimensional instability of Invar alloy was bad.

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## 1. Introduction

The most prominent characteristic of Invar alloy (4J36) is its very low or even zero thermal expansion coefficient in a wide temperature range [1]. This characteristic of Invar alloy also has a high degree of dimensional instability [2-4]. Because of these advantages, Invar alloy has a wide application in the field of precision instrument [5-6]. With the further development of the technology, the accuracy requirements for precision instrument are also increasing and the existing heat treatment process for Invar alloy sometimes can't meet the requirements for the dimensional instability of more high-precision instrument [7-8]. Some experts reported that the dimensional instability of materials were related to both the coefficient of thermal expansion and internal residual stress [9-12]. Therefore, in this article, the effect of aging process on the coefficient of thermal expansion and residual stress of invar alloys was studied in detail.

Micro-changes in the size of metal structure will occur under natural conditions and it is the result of two mainly factors. (1) creeping caused by the mechanical processing before using and the internal residual stress generated when using. (2) a series of instable factors, such as phase transformation, dislocation sliding and so on. The phase transition and dislocation sliding on the microstructure depend on both the internal structure and heat treatment condition of material. If the internal organization of material is stable, the dimensional instability of material also depend on the relaxation of residual stress inside the material[13-16]. For example,

during the high-temperature deformation process of some deforming alloys, a part of the internal organization in the alloys would be metastable phase which was caused by intense deformation. During the cooling process from high temperature, internal stress would continue to increase in material and the internal stress would relax after a period of time. In this process, the internal stress played an important role in promoting phase transition and the change of organization. Therefore, micro-strain generated on the macro-structure and it made the internal organization of material become stable phase [17].

## 2. Experimental

The Invar alloys used in this article were forged by Dongbei Special Steel Group Co., Ltd. The alloy samples were rod-like after vacuum annealing. Table 1 shows the chemical composition of the Invar alloys.

*Table 1. Chemical compositions of Invar alloy (wt%).*

Ni	Co	Mn	Si	C	Fe
36.14	0.52	0.28	0.04	0.014	balanced

During the heat treatment process, the samples with the size of  $\Phi 6 \times 25$ mm were vacuum sealed (the degree of

vacuum is  $10^{-1}$ Pa). The Invar alloys were quenched at  $820^{\circ}\text{C}$  and the holding time was 20min. Then, the alloys were respectively aged at the temperature ranging from  $200^{\circ}\text{C}$  to  $500^{\circ}\text{C}$  and the aging time was 2 hours. Finally, the effect of aging process on the coefficient of thermal expansion and residual stress of Invar alloys was analyzed.

Residual stress was measured by XSTRESS3000 stress analyzing instrument. The measurement was based on Chinese national standard (GB/T7704-2008 "X-ray stress measurement method"). In the measurement, Cr target was used, collimator was 2mm, voltage was 2kV and current was 6.7mA. Both ends of the samples were electrolytic polished. At the ends of the samples, three sets of data and their average value were measured.

The coefficients of thermal expansion of different samples were measured by NETZSCH DIL 402C dilatometer made by Germany NETZSCH Company. The resolution of the dilatometer could reach 8nm and the test accuracy was  $10^{-8}$ . The samples were cylindrical with the size of  $\Phi 6 \times 25\text{mm}$ . The test temperature is  $-40 \sim 120^{\circ}\text{C}$  and the heating rate was 5K/min.

Microstructure was observed by Axiovert 40 MAT Zeiss optical microscope, the FEI-Sirion field emission scanning electron microscope and TENCNAI G2 F30 transmission electron microscope made by Philips Company with the accelerating voltage of 200kV. The TEM samples were prepared by electrolysis dual-jet thinning instrument.

### 3. Results and discussion

#### 3.1 Microstructure analysis

Fig. 1 shows both the Invar alloys in original state and the alloys prepared by solution treatment and quenching. The organization of the alloys was uniform. There was no obvious defect inside the material and the grains had no obvious texturing. The size of the grains in original Invar alloys was  $40\mu\text{m}$  (Fig. 1a)). The size of the grains prepared by quenching was about  $20\mu\text{m}$  (Fig. 1b)).

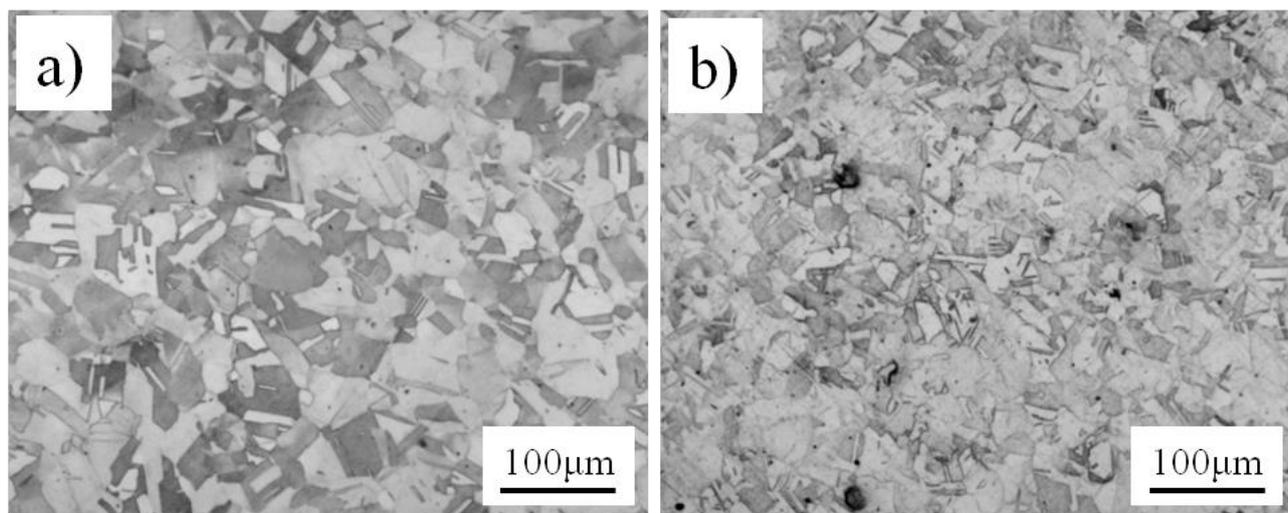


Fig. 1. The microstructure of original Invar alloys; a) original Invar alloys; b) the Invar alloys prepared by quenching.

Fig. 2 shows the microstructure of the alloys at different aging temperatures. The organization of the alloys was uniform and no obvious defects were observed. It is obvious that the grain size of Invar alloy increased with the increasing of the aging temperature. The grain size of the Invar alloy only prepared by quenching at  $820^{\circ}\text{C}$  was similar with the Invar alloy aged at  $200^{\circ}\text{C}$  after quenched, as shown in Fig. 3a).

However, the grain size of the Invar alloy aging at  $300^{\circ}\text{C}$  and  $400^{\circ}\text{C}$  was a bit larger (about  $25\mu\text{m}$ ). When the aging temperature became  $500^{\circ}\text{C}$ , the grain size of the alloy increased significantly (about  $35\mu\text{m}$ ) and its grain size was a bit smaller than original Invar alloys.

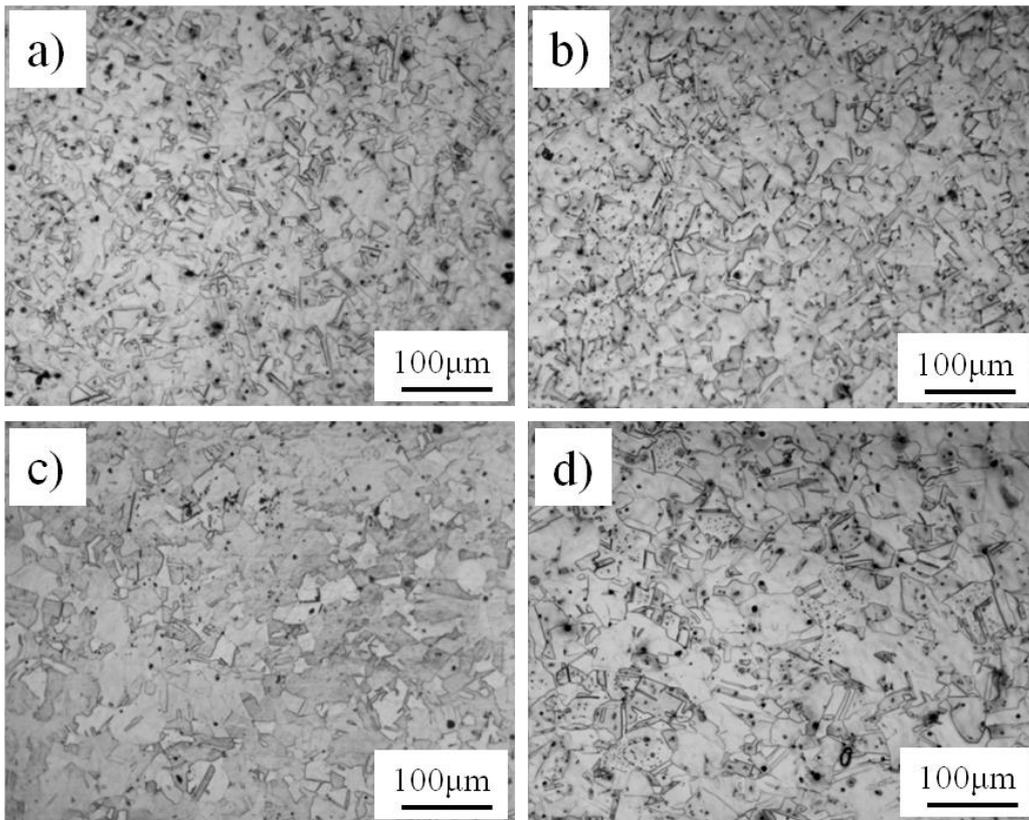


Fig. 2. The microstructure of the alloys at different aging temperatures; a) 200°C; b) 300°C; c) 400°C; d) 500°C.

Fig. 3 shows the TEM observation of Invar alloy. The organization could be identified as austenite phases with fcc structure by the diffraction spots in Fig. 3a) and b).

Twinning organizations also could be observed during the deformation process of the alloys.

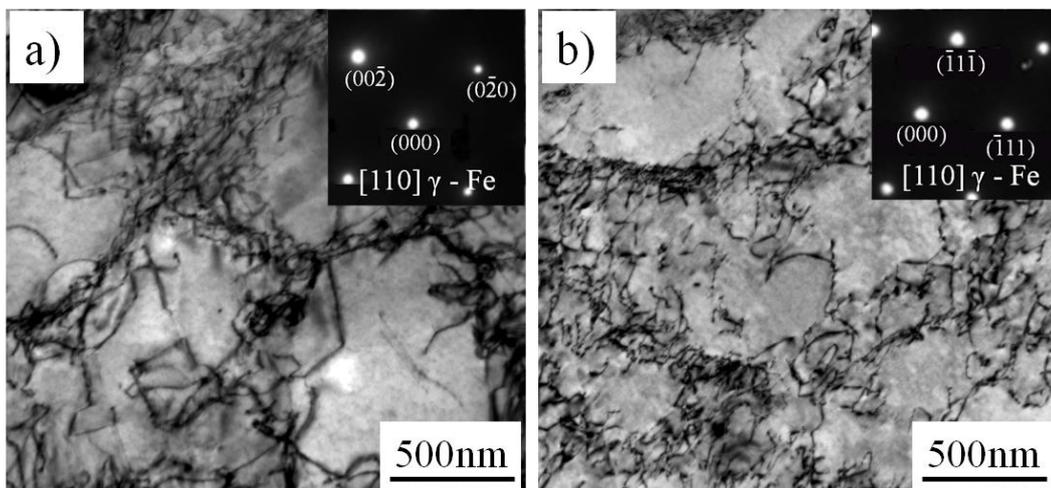


Fig. 3. The microstructure of Invar alloy in original state.

In order to get the best process of heat treatment which could make the coefficient of thermal expansion

and residual stress of the alloys lowest, different processes of heat treatment were used at the temperature ranging

from 200°C to 500°C. Fig. 4 shows the TEM photos of the Invar alloy aged at 200°C, 300°C, 400°C and 500°C after quenched at 820°C. The dislocation density of Invar alloy

only prepared by quenching at 820°C is very high and it changed significantly after aged (Fig. 4a).

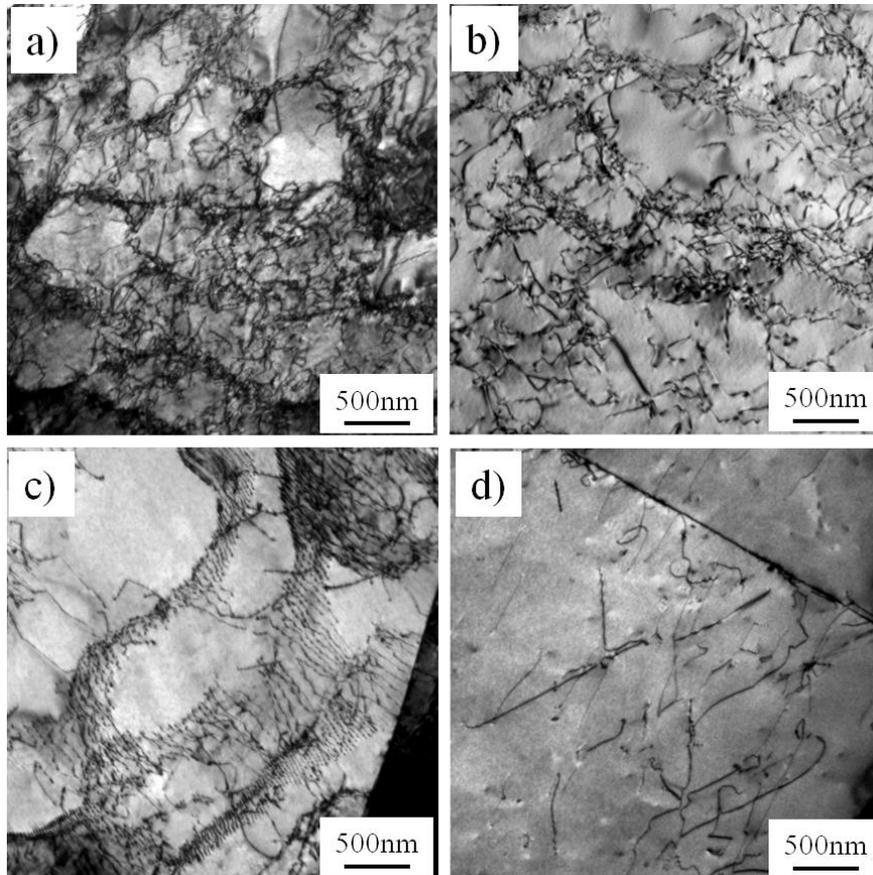


Fig. 4. The TEM photos of the Invar alloy after aged; a) 200°C; b) 300°C; c) 400°C; d) 500°C.

The dislocation density decreased significantly after aged at 300°C (Fig. 4b)). After aged at 400°C, the dislocation density of the alloys also decreased compared with aged at 300°C and the form of dislocations changed from mutual entanglement to parallel state (Fig. 4c)). This change was the precursor to the formation of sub-grain. The dislocation density decreased quickly and the sub-grain grew significantly after aged at 500°C (Fig. 4d)). At that time, there were only a few of dislocations in the alloys.

### 3.2 Residual stress

Fig. 5 shows the residual stress of the Invar alloys quenching at 820°C and aged at different temperature. Original Invar alloys were treated by high-temperature vacuum annealing. Therefore its residual stress which was compressive stress was only 50MPa. After quenched at 820°C, the residual compressive stress of the Invar alloys which was enhanced greatly reached 349.1Mpa.

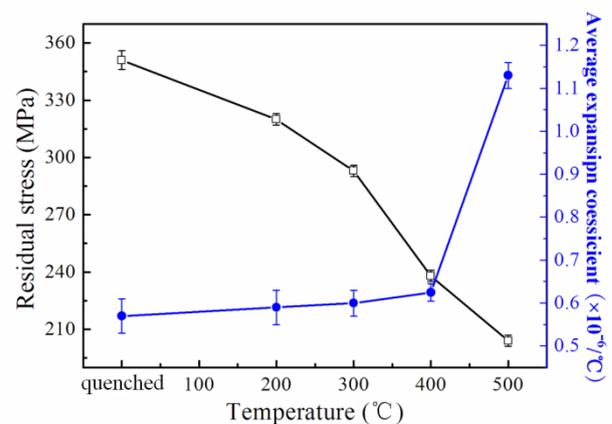


Fig. 5. Residual stress and average coefficient of thermal expansion of invar alloys with different aging temperature.

The residual stress of the Invar alloy decreased significantly with the increase of the aging temperature. The decrease of the residual stress caused by the change of the temperature would enhance with the increase of the aging temperature. The residual stress in the alloys decreased with the increase of aging temperature.

$$\sigma_s = \sigma_0 + Kd^{-\frac{1}{2}} \quad (1)$$

Hall-Petch formula [18] could be expressed as Eq. (1). In Eq. (1),  $\sigma_s$  donates yield stress,  $K$  a constant related to crystal type and  $d$  grain size. According to Eq. (1), the stress concentration caused by dislocation pile in large grains could have more chances to cause micro-plastic deformation in the adjacent grains than small grains. The smaller grain size in per unit volume of the alloys was, the larger the grain boundary area was and the greater the obstacles to dislocation motion were. Therefore, the smaller the grain was, the larger yield strength and the internal stress of the alloy were. Therefore, not only a single grain size, but also other organizational structure and stress relations should be considered when analyzing. Generally, both of the two factors could cause the change of the grain size for the alloys with metastable organization. Phase transition and organizational change would enhance the relaxation of internal residual stress in materials and the changes in the internal stress also played an active role in the phase transition and organizational change at the same time. Therefore, the material would reach a state of equilibrium.

### 3.3 Coefficient of thermal expansion

Fig. 6 shows the average coefficients of thermal expansion of the original Invar alloys, the alloys quenched at 820°C and the alloys aged at 200°C, 300°C, 400°C and 500°C, respectively. The average CTE of the original Invar alloy and the alloy aged at 500°C were much larger than the others. At the temperature ranging from -40°C to 0°C, the average coefficients of thermal expansion of the alloys aged at 200°C and 300°C were smaller than the alloys quenched at 820°C. At the temperature ranging from 0°C to 75°C, the average coefficients of thermal expansion of the alloy samples except the original Invar alloy and the alloy aged at 500°C were similar (about  $0.6 \times 10^{-6}/^\circ\text{C}$ ). At the temperature ranging from 75°C to 110°C, the average coefficients of thermal expansion of the alloys aged at 200°C, 300°C and 400°C were larger than the alloy quenched at 820°C. Compared with the alloys quenched at 820°C, the average coefficient of thermal expansion of the alloys aged at 500°C ( $1.2 \times 10^{-6}/^\circ\text{C}$ ) increased 100%.

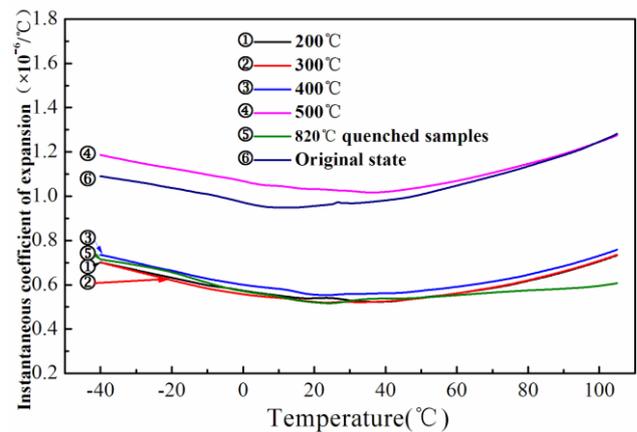


Fig. 6. The average coefficients of thermal expansion of all the alloy samples.

Meanwhile, the coefficient of thermal expansion of invar alloy is minimal near room temperature. On heating from absolute zero to the Curie point, the characteristic decreases in atomic spacing which is superimposed upon the normal dilation due to the rising temperature. These two opposing effects happen to counter-balance each other almost completely in the vicinity of room temperature and thereby result in a low expansion coefficient [19]. The competition of two opposing effects may cause the minimum of thermal expansion coefficient too.

Fig. 5 shows the average coefficient of thermal expansion of invar alloy at the temperature ranging from 20°C to 75°C. The increase of tempering temperature could enhance the average coefficients of thermal expansion of the alloys. After aged at 500°C, the average coefficients of thermal expansion of the alloys increased 100% and the residual stress decreased. These changes proved that the relaxation of the residual stress inside the Invar alloy would cause the increase of its average coefficients of thermal expansion. Therefore, there was no characteristic of low expansion in the Invar alloy after aged at 500°C. In order to assure the dimensional instability of invar alloy, 400 °C was optimum aging temperature.

### 4. Conclusions

Compared to original state, the residual stress of invar alloy quenched increase significantly. However, the residual stress of the Invar alloy decreased with the increase of the aging temperature. The coefficients of thermal expansion of the Invar alloy did not change obviously with the increase of the aging temperature when the aging temperature was below 400°C. However, the coefficients of thermal expansion increased significantly when the aging temperature was 500 °C. In order to assure the dimensional instability of invar alloy, 400 °C was optimum aging temperature.

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