

## Microstructural Evolution and High Strain Rate Mechanical Property of Cobalt Superalloy Contain Titanium

(Evolusi Mikrostruktur dan Sifat Mekanik Kadar Terikan Tinggi Superaloi Kobalt yang mengandungi Titanium)

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### ABSTRACT

*The high strain rate properties and microstructure observations of cobalt base superalloyd contains 0.9% Ti were investigated using Hopkinson bar. These cobalt base superalloys are tested at strain rates of  $2 \times 10^3$ ,  $3 \times 10^3$  and  $4 \times 10^3$  s<sup>-1</sup> and at different temperatures (25, 500 and 900°C), respectively. It is found that the stress flow and strain rate sensitivity increases with increasing strain rate but decrease with increasing temperature. The microstructure observations confirm that the high strain rate mechanical behavior of the cobalt base superalloys specimens are directly related to the effects of the strain rate, temperature and the evolution of the microstructural texture. The strengthening mechanism in cobalt base superalloys is the multiplication of dislocation. The dislocation density increases with increasing strain rate but decrease with increasing temperature.*

*Keywords: Cobalt superalloy; dislocation; strain rate; Ti additions*

### ABSTRAK

*Pemerhatian sifat kadar terikan yang tinggi dan mikrostruktur superaloi asas kobalt yang mengandungi 0.9% Ti dikaji dengan menggunakan bar Hopkinson. Superaloi berasaskan kobalt ini diuji pada kadar terikan  $2 \times 10^3$ ,  $3 \times 10^3$  dan  $4 \times 10^3$  s<sup>-1</sup> dan masing-masing pada suhu yang berbeza (25, 500 dan 900C). Adalah didapati bahawa kepekaan tegasan aliran dan kadar terikan meningkat dengan meningkatnya kadar terikan tetapi menurun dengan pertambahan suhu. Pemerhatian mikrostruktur mengesahkan bahawa kelakuan mekanik kadar terikan yang tinggi oleh spesimen superaloi berasaskan kobalt berkait rapat dengan kesan kadar terikan, suhu dan evolusi tekstur mikrostruktur. Mekanisme pengukuhan dalam superaloi berasaskan kobalt adalah pendaraban perkelehannya. Ketumpatan perkehelan meningkat dengan peningkatan kadar terikan tetapi berkurangan dengan pertambahan suhu.*

*Kata kunci: Kadar terikan; penambahan Ti; perkelehan; superaloi kobalt*

### INTRODUCTION

In recent years, the cobalt base superalloys become the suitable material for use in nuclear and aerospace industry due to their excellent corrosion resistance in hot environment, wear resistance and fatigue properties (Agarwal & Ocken 1990; Kuzucu et al. 1998; Rao et al. 1997; Suzuki et al. 2007; Tang et al. 2008). The strengthening mechanisms of cobalt base superalloys are mainly by utilizing the carbide precipitates formed in cobalt alloy matrix and grain boundary, such as  $M_6C$  and  $M_{23}C_6$  and solid solution strengthening elements, such as tungsten, molybdenum and tantalum (Alauddin et al. 1996; Yadav & Ramesh 1995). However, the high temperature strength and ductility of cobalt base superalloys are inferior to those of nickel base superalloys. Sato et al. (2006) have reported that a ternary compound,  $Co_3Ta$  and  $Co_3Ti$  with  $L_{12}$  structure was developed to strengthen the cobalt base superalloys. The novel cobalt base superalloys were strengthened by high volume fractions of the  $L_{12}$  compound leads high

mechanical properties, superior hot corrosion and wear resistance compared with nickel base superalloys for the applications in severe environments. In general, the flow stress in engineering materials increases with the increasing strain rate, but decreases with the increasing temperature. The effects of strain rate in the particular material are important.

The deformation behaviour and microstructural evolution of Co-base superalloy under quasi-static loading conditions have been extensively examined (Krol et al. 2004). However, the literature contains scant information regarding the high strain rate deformation behavior of cobalt base superalloy. Accordingly, the present study utilises a compressive split-Hopkinson pressure bar (SHPB) system to investigate the high strain rate deformation behavior and microstructural evolution of Co-base superalloy specimens deformed at room temperature conditions under strain rates ranging from  $2 \times 10^3$  to  $4 \times 10^3$  s<sup>-1</sup>. The effects of the strain rate on the flow response and dislocation substructure were systematically

explored. Moreover, the relationship between the flow stress and the dislocation density was examined and discussed. Previous literatures have shown that the rate of dislocation multiplication and the dislocation structures themselves were highly sensitive not only to the deformation temperature and strain rate, but also to the original crystal structure of the material. In general, the rate of dislocations multiplication increases with increasing strain rate and leads to an improved material strength (Albert & Gray III 1997; Jarmakani et al. 2007; Meyers et al. 1995). This study investigated the effects of the addition of titanium on the mechanical properties and microstructure evolution of cobalt base superalloy and the flow stress behaviors of the superalloy at room temperatures. The microstructure observations of the tested specimens were examined using transmission electron microscopy (TEM). The stress-strain relation and the evolution of the microstructure were also discussed.

#### EXPERIMENTAL DETAILS

The cobalt based superalloys were prepared in a vacuum arc melted furnace. The mass chemical composition of the cobalt base superalloy (as measured by a glow discharge spectrometer (GDS)) were 18 Cr, 10 Ni, 13 W, 1.2 Mn, 0.04 C and the balance Co, with contents of 0.9 Ti. The solution heat treatment of the ingots was carried out at 1300°C for 2 h in a vacuum furnace. Then they were aged at 680°C for 4 h and at 850°C for 20 h and poured into a heated permanent metal mold. Then an indirect extrusion machine forms bars with dimensions of 13 × 700 mm (diameter × length). Finally, these bars were machined into cylindrical specimens with a length and diameter of 9.7 mm and the ends of each specimen were then carefully finished using a grinder. In the dynamic tests, the specimens were deformed under room temperature conditions (25°C) at strain rates of 2000, 3000 and 4000 s<sup>-1</sup>, respectively, using a compressive SHPB system. The striker bar, incident bar and transmitter bar of the SHPB system were all machined from SKD11 die steel bar with a diameter of 12.5 mm. The incident and transmitter bars each had a length of 1 m, while the striker bar had a length of 367 mm. In performing the tests, the specimens were sandwiched between the incident bar and the transmitter bar and the incident bar was then impacted by the striker bar (fired by a gas gun).

After the high strain rate tests, the deformed specimens were examined via optical microscopy (OM) and were examined using a JEOL TEM-3010 transmission electron microscope (TEM) operating at 300 kV. The specimens for TEM observation were prepared by cutting thin foils of 350 μm thickness from the deformed specimens. The disks were then punched from each foil, finally ground to a thickness of 200 μm and then twin-jet polished with a solution of Spar etchant (100 mL of distilled water, 100 mL of 32% HCl, 10 mL of 65% HNO<sub>3</sub> and 0.3 mL of 1-methoxy-2-propanol).

## RESULTS AND DISCUSSION

#### STRESS-STRAIN CURVE RESPONSE

Figure 1(a)-1(c) shows the mechanical behavior of stress-strain curves for cobalt base superalloy which contains 0.9% Ti deformed at different temperature of 25, 500 and 900°C and strain rates of 2×10<sup>3</sup> s<sup>-1</sup>, 3×10<sup>3</sup> s<sup>-1</sup> and 4×10<sup>3</sup> s<sup>-1</sup>, respectively. It can be observed that the flow stress of Co-0.9 Ti superalloy increases with strain rate and strain but decreases with increasing temperature. In general, it was noted that all three curves had a monotonic parabolic characteristic; indicating the occurrence of strain hardening at higher strain rates. Furthermore, it was observed that for each strain rate, the flow stress increased gradually with an increasing strain, while for a constant strain, the flow stress increases gradually with an increasing strain rate. However, under a fixed strain rate, a decrease of stress with increasing temperature is observed. In addition, it is seen that the strain rate affects not only the flow stress, but also the fracture strain. For example, none of the specimens fractured when deformed at a strain rate of 2×10<sup>3</sup> s<sup>-1</sup>, while all of the specimens fractured when tested at a higher strain rate of 3×10<sup>3</sup> s<sup>-1</sup> or 4×10<sup>3</sup> s<sup>-1</sup>. It was noted that the fracture strain increased with an increasing strain rate. Thus, it was inferred that the ductility and strength of Co-0.9Ti superalloy increases with an increasing strain rate.

As discussed by Lee et al. (2010), the flow stress-strain data shown in Figure 1 can be fitted using a power law hardening equation with the form  $\sigma_2 = A + B\epsilon^n$ , where A is the yield strength; B is the material constant; and n is the work hardening coefficient. Table 1 presents the results obtained for A, B and n for the present Co-base superalloy contain Ti at each of the considered strain rates and temperatures. It was seen that the yield strength, material constant and work hardening coefficient all increase with the increasing strain rate but decrease with increasing temperature. This finding suggested that the dislocation density and multiplication rate increased at higher strain rates; causing a corresponding increase in the resistance of the Co superalloy contain Ti to plastic flow. But the temperature causes the inhibition of the dislocation.

#### STRAIN RATE EFFECT

It was seen that the flow stress of the cobalt base superalloys contained Ti increase with the increasing strain rate. The effect of the strain rate on the stress can be quantified via the strain rate sensitivity parameter  $\beta$ . The strain rate sensitivity,  $\beta$ , of the cobalt superalloy can be calculated from the experimental data presented in Figure 1 and the equation as shown in Ezz et al. (1995):

$$\beta = (\partial\sigma / \partial \ln \dot{\epsilon}) = \frac{\sigma_2 - \sigma_1}{\ln(\dot{\epsilon}_2 / \dot{\epsilon}_1)}, \quad (1)$$

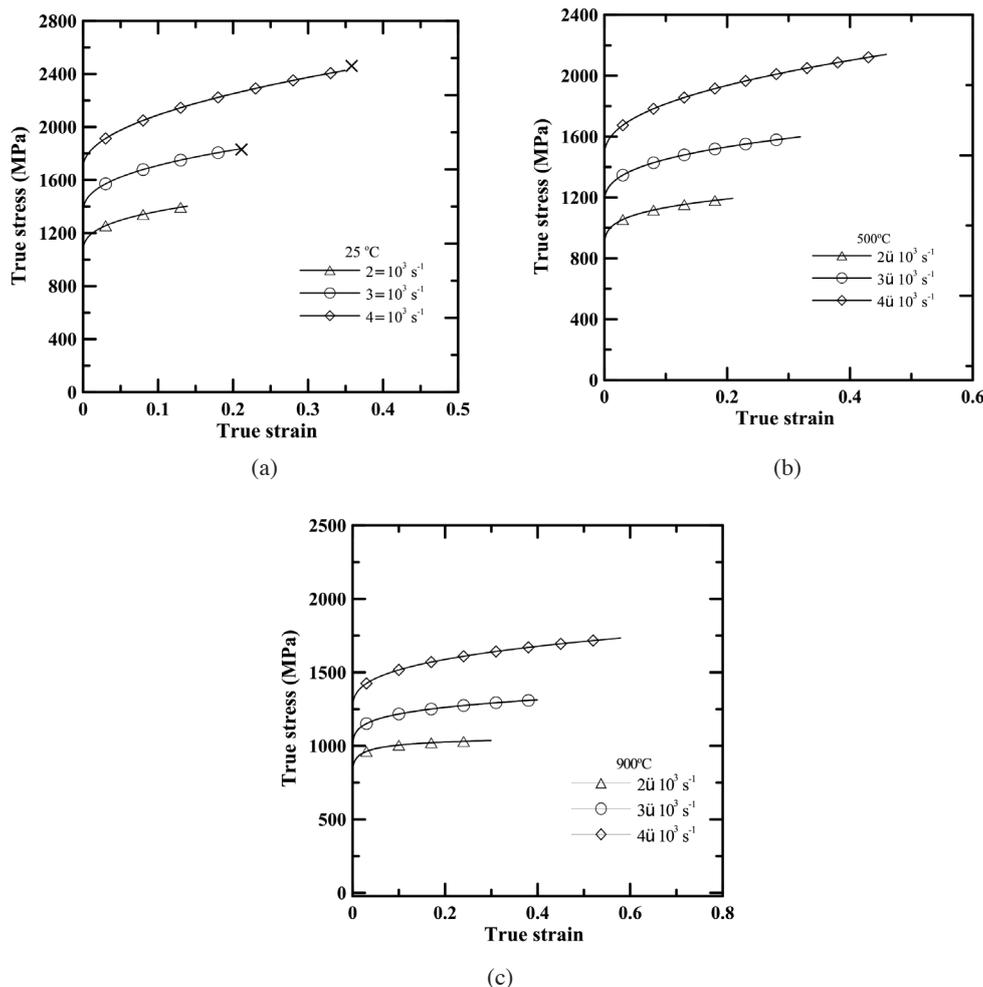


FIGURE 1. True stress-strain curves of cobalt base superalloy deformed at strain rates of  $2 \times 10^3 \text{ s}^{-1}$ ,  $3 \times 10^3 \text{ s}^{-1}$  and  $4 \times 10^3 \text{ s}^{-1}$  and temperatures of  $25^\circ\text{C}$ , respectively

TABLE 1. Mechanical properties of Co-base superalloy impacted at different high strain rates

T ( $^\circ\text{C}$ )	Strain rate ( $\text{s}^{-1}$ )	Yield stress A (MPa)	Material constant B (MPa)	Work hardening coefficient n
25	2000	1040	710	0.34
	3000	1350	915	0.40
	4000	1695	1230	0.49
500	2000	840	540	0.3
	3000	1200	680	0.36
	4000	1480	950	0.42
900	2000	545	565	0.12
	3000	910	505	0.25
	4000	1250	610	0.39

where the flow stresses  $\sigma_2$  and  $\sigma_1$  were obtained from tests conducted at strain rates of  $\dot{\sigma}_2$  and  $\dot{\sigma}_1$ , respectively and were calculated at the same value of plastic strain. Figure 2 presents the strain rate sensitivity of the Co-0.9Ti superalloy as a function of strain. It can be seen that the strain rate sensitivity of the Co-0.9Ti superalloy increased with strain rate but decreased with increasing temperature.

#### MICROSTRUCTURAL OBSERVATIONS AND ANALYSIS

Figure 3 presents the OM micrograph of undeformed Co-0.9Ti superalloy and it shows the presence of a random distribution of annealing twin. Figures 4(a)-4(c) presents the TEM images showing the dislocation substructures of the specimens deformed at  $25^\circ\text{C}$  and strain rates of  $2 \times 10^3 \text{ s}^{-1}$ ,  $3 \times 10^3 \text{ s}^{-1}$  and  $4 \times 10^3 \text{ s}^{-1}$ , respectively. As shown in Figure 4(a), the microstructure of the specimen deformed

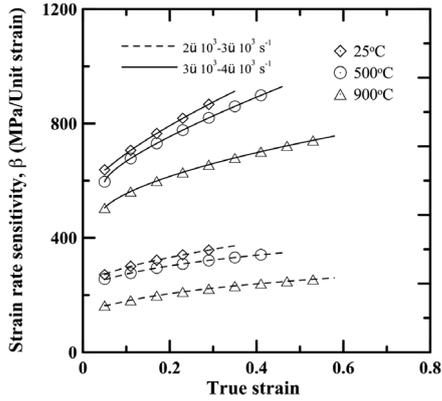


FIGURE 2. Variation of strain rate sensitivity as function of strain Co-base superalloy at temperature of 25°C

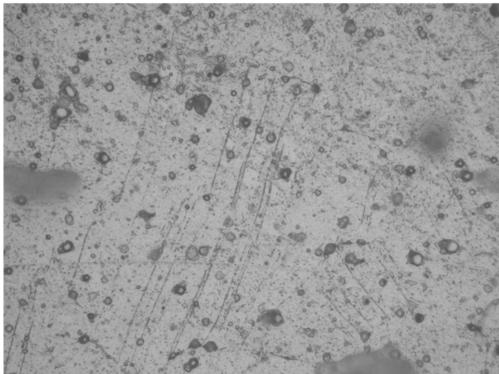


FIGURE 3. Optical electron micrographs of undeformed cobalt base superalloy at temperatures of 25°C

at  $2 \times 10^3 \text{ s}^{-1}$  consists of loosely-tangled dislocation cells. It was noted that the dislocation density was higher than that of the un-deformed microstructure. The dislocation density increased further as the strain rate was increased to  $3 \times 10^3 \text{ s}^{-1}$  (Figure 4(b)). Moreover, an accumulation of the dislocations at the grain boundaries was observed together with the formation of small dislocation cells with thin walls. The increased dislocation density prompts the formation of a tangled dislocation structure, which acts as an obstacle to subsequent dislocation motion. As the strain rate is further increased to  $4 \times 10^3 \text{ s}^{-1}$  (Figure 4(c)), the dislocation density and number of tangled dislocations both significantly increased. The dense arrangement of tangled dislocations hinders the further motion of dislocations within the deformed microstructure and therefore enhanced the resistance of the cobalt base superalloy to plastic flow (as shown in the stress-strain curves presented in Figure 1). Furthermore, it was known that cobalt base superalloy contains Ti, formation in the coherent precipitate of  $\text{Co}_3\text{Ti}$  compound which can also enhanced the strength of the cobalt base superalloy. Moreover, the twin was also the enhanced mechanism of Co-base superalloy. The twin microstructures can be seen from Figures 4(a)-4(c). Figure 5(a)-5(d) shows the TEM micrographs of the Co-0.9Ti superalloy deformed at temperature of 500°C and 700°C under strain rates of  $2 \times 10^3 \text{ s}^{-1}$  and  $4 \times 10^3 \text{ s}^{-1}$ , respectively. It can be seen that specimens deformed at 500°C and  $2 \times 10^3 \text{ s}^{-1}$ , Figure 5(a), a rapid annihilation of dislocations occurred and larger dislocation cell have been formed. When the strain rate increased to  $4 \times 10^3 \text{ s}^{-1}$  and at the same temperature of 500°C, Figure 5(b), the density and the tangles of dislocations were increased. Comparing the

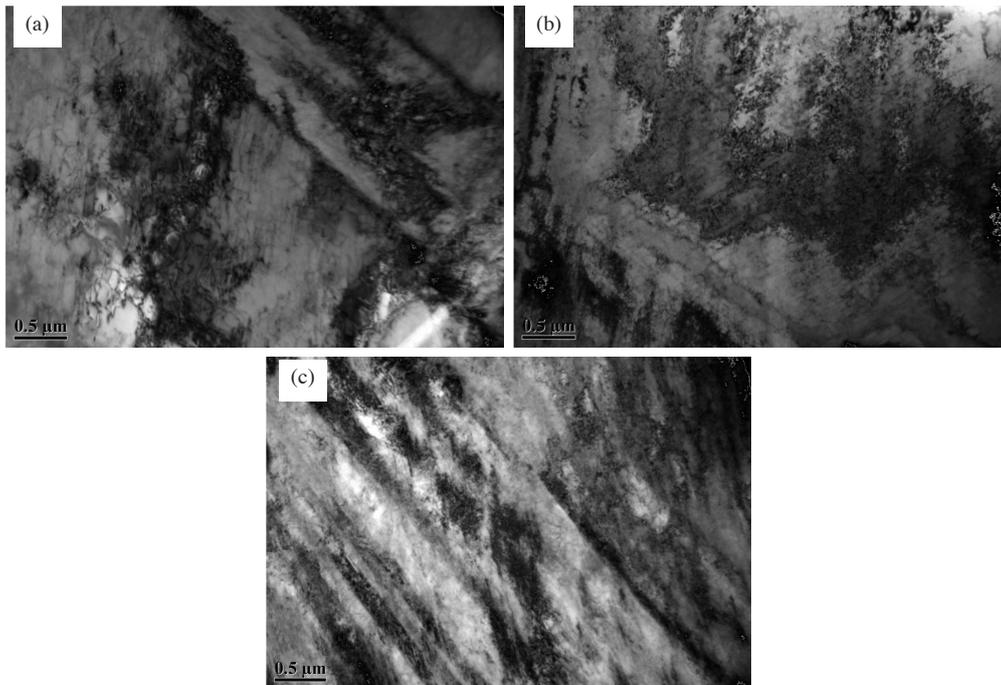


FIGURE 4. TEM micrographs of dislocation microstructures of specimens deformed at temperature of 25°C and strain rates of (a)  $2 \times 10^3 \text{ s}^{-1}$ , (b)  $3 \times 10^3 \text{ s}^{-1}$  and (c)  $4 \times 10^3 \text{ s}^{-1}$

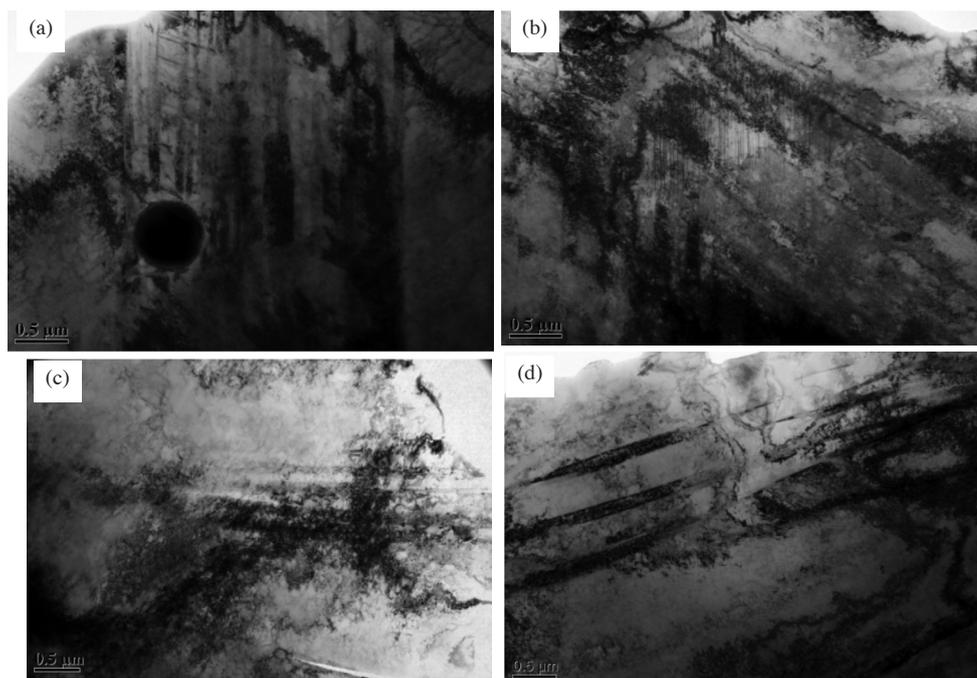


FIGURE 5. TEM micrographs of dislocation microstructures of specimens deformed at temperature of 500°C and strain rates of (a)  $2 \times 10^3 \text{ s}^{-1}$ , (b)  $4 \times 10^3 \text{ s}^{-1}$ ; and temperature of 900°C and strain rates of (c)  $2 \times 10^3 \text{ s}^{-1}$  and (d)  $4 \times 10^3 \text{ s}^{-1}$

results with 25 and 500°C, the effect of strain rate on dislocation structure at 900°C is similar, but the density and interaction of dislocations were decreased (Figure 5(c) and 5(d)).

#### CONCLUSION

The mechanical response and microstructural evolution of Co-base superalloy have been investigated under room temperature conditions at strain rates of  $2 \times 10^3$ ,  $3 \times 10^3$  and  $4 \times 10^3 \text{ s}^{-1}$ , respectively, using a compressive split-Hopkinson pressure bar system. The results have shown that the deformation behaviour of Co-base superalloy highly sensitive to the strain rate. Specifically, the flow stress and work hardening coefficient both increase significantly with increasing strain rate. Moreover, the strain rate sensitivity was found to increase with the increasing strain rate. TEM observations have shown that the dislocation density increases with increasing strain rate, but decreases with the increasing temperature. The high dislocation density and small dislocation cell size result in an increased flow stress. Furthermore, the cobalt base superalloy has the Ti contents, formation the coherent compound of  $\text{Co}_3\text{Ti}$  which can also enhanced the strength of the cobalt base superalloy. Moreover, the twin can also be observed in all the deformed conditions.

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