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Duplex Precipitates and Their Effects on the Room-temperature Fracture Behaviour of a NiAl-Strengthened Ferritic Alloy

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Duplex precipitates are presented in a NiAl-strengthened ferritic alloy. They were characterized by the ultra-small angle X-ray scattering and transmission electron microscope. Fine cooling precipitates with the size of several to tens of nanometres harden the matrix considerably at room temperature. Cracks are likely to initiate from precipitates, and coalesce and propagate quickly through the matrix due to the excessive hardening effect of cooling precipitates, which lead to the premature fracture of NiAl-strengthened ferritic alloys.

Keywords: NiAl-strengthened Ferritic Steels, Small-angle X-ray Scattering (SAXS), Precipitation, Fracture, Order Strengthening

NiAl-strengthened ferritic alloys have received considerable attention due to their good high-temperature creep resistance for applications in fossil-energy power plants.[1,2] The small lattice mismatch between the α -iron matrix and NiAl-type precipitates effectively delays the precipitate-coarsening kinetics at elevated temperatures ($\sim 700^\circ\text{C}$).[3,4] Studies have been performed to elucidate the creep mechanisms of NiAl-strengthened ferritic alloys.[5–9] However, a lack of the room-temperature ductility limits applications of NiAl-strengthened ferritic alloys.[6,10–13] Factors such as the precipitate-distribution parameters [6,10,11,13] (e.g. the precipitate volume fraction, f , average size, $\bar{r}$, and inter-particle distance, L) and solute atoms like aluminium and nickel in the matrix [11] were discussed. The room-temperature fracture mechanism of NiAl-strengthened ferritic alloys is not yet understood. Previous studies on NiAl-strengthened ferritic alloys revealed the presence of duplex precipitates (aged and fine cooling ones).[6,14] However, effects of fine cooling precipitates have not been investigated. In this study, we characterize duplex precipitates by the ultra-small angle X-ray scattering (USAXS) [15–17] and transmission electron microscope (TEM), and discuss their

effects on the room-temperature fracture behaviour of NiAl-strengthened ferritic alloys.

A model NiAl-strengthened ferritic alloy, designated as FBB8, was investigated. An ingot with the size of $\sim 12.7 \times 25.4 \times 1.9$ cm was prepared by the Sophisticated Alloys, Inc., using the vacuum-induction melting. The nominal and measured compositions of the ingot are given in Table 1. The hot iso-thermal pressing ($1,200^\circ\text{C}/103$ MPa/4 h) was conducted on the ingot to minimize casting porosity. Encapsulated specimens (in a quartz tube with the vacuum environment) were first solution treated at $1,200^\circ\text{C}$ for 1 h, followed by air cooling (AC), and then, aged in the temperature range from 700°C to 950°C . The water quenching (WQ) and AC were used, following the age treatment. After the solution treatment (ST), precipitates with the size of 36 ± 5 nm were observed.[4] The nucleation and growth of NiAl-type precipitates are too fast to be suppressed by WQ, following ST.[4] The final grain size is ~ 100 μm . A small amount of segregation enriched in Zr is found along grain boundaries and within grains.

Readers are referred to Ref. [4,16] for the experimental details of USAXS. The size of cooling precipitates is assumed to follow the Gaussian distribution. The

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Table 1. The compositions of the FBB8 ingot, weight per cent.

	Al	Ni	Cr	Mo	Zr	B	Fe
Nominal composition	6.5	10	10	3.4	0.25	0.005	Bal.
Measured composition	6.66	10.29	10.30	3.57	0.25	0.005	Bal.

form factor for spherical particles is employed in data modelling. Note that the contrast between the matrix and precipitates in USAXS originates from the composition difference (especially the aluminium content). The TEM images were taken, using a Zeiss Libra 200 model. Discs with the diameter of 3 mm were polished to the thickness of $\sim 70 \mu\text{m}$, and were further thinned in a Fischione twin-jet electropolisher, followed by ion milling with the energy of 3.5 keV for about 10 minutes. The electrolyte of the 5 volume-per cent HCl in ethanol was used at room temperature with the current of 6–8 μA .

Room-temperature tension/compression tests were conducted at the strain rate of 10^{-4} s^{-1} , using the hydraulic Materials Testing System machines. The 0.2% yield stress, σ_Y , was derived from three compression tests. The Vickers hardness of specimens was measured. At least six measurements were performed for each specimen. The fractured specimens were studied by scanning electron microscopy (SEM). Some deformed specimens were examined by TEM to reveal the deformation mechanisms.

Figure 1(a) and 1(b) shows the USAXS scattering curves, documenting the bimodal precipitate distribution in specimens aged for 100 h at 800°C, together with the corresponding TEM images. In this paper, the subscript of ‘1’ stands for aged precipitates and ‘2’ for cooling precipitates. Since the scattering vector, q , is inversely related to the real dimension of precipitates,[15] small precipitates show as features in USAXS curves at large q values. For example, in Figure 1(a), aged precipitates ($\bar{r}_1 \sim 166 \text{ nm}$ [4]) are represented by features (the Guinier knee and subsequent power-law slope) in the q range from 0.0003 to 0.02 $\text{\AA}^{-1}$, while features from cooling precipitates ($\bar{r}_2 \sim 3.1 \pm 0.5 \text{ nm}$) dominate the q regime from 0.02 to 0.15 $\text{\AA}^{-1}$. Beyond 0.15 $\text{\AA}^{-1}$, the scattering intensity from the background overwhelms that from precipitates. By comparing Figure 1(a) and 1(b), the size of cooling precipitates is strongly related to the cooling rate, following the ageing treatment. $\bar{r}_2 \sim 3.1 \pm 0.5$ and $7.6 \pm 1.8 \text{ nm}$ for WQ and AC, respectively, following the ageing treatment at 800°C. It is reported that the solubility of aluminium and nickel in α iron decreases, as the temperature decreases in the Fe–Ni–Al system.[18] Consequently, NiAl-type precipitates are likely to nucleate and grow in the cooling process due to supersaturated aluminium and nickel atoms in the matrix, and it is confirmed by the size dependence of fine precipitates on the cooling rate following the ageing

treatment. The corresponding TEM images demonstrate that both aged and cooling precipitates have a B2-ordered structure (Figure 1(b)). The cooling precipitates-free zone around aged precipitates is observed. Specimens aged for a longer time at 800°C show a similar size distribution of cooling precipitates. $\bar{r}_2$ also presents the dependence on the ageing temperatures, as can be seen in Figure 1(c). Features of cooling precipitates for specimens aged at 700°C cannot be directly captured by USAXS due to the background scattering in the q regime greater than 0.1 $\text{\AA}^{-1}$. When these specimens were aged for 50 h at 500°C, fine precipitates show their features in the relatively small q area (0.05–0.2 $\text{\AA}^{-1}$), as shown in Figure 1(d). The double-ageing treatment probably does not change the volume fraction of fine precipitates because of the high precipitation rate of NiAl-type precipitates during cooling. Teng et al. [14] observed cooling precipitates ($\bar{r}_2 \sim 1.5 \text{ nm}$) through the atom probe tomography for the FBB8 specimen aged for 100 h at 700°C + AC. The composition of these cooling precipitates was determined to be $\text{Ni}_{26.3}\text{Al}_{41.6}\text{Fe}_{26.9}\text{Cr}_{3.3}\text{Mo}_{1.7}$, which has an aluminium content similar to that of aged precipitates ($\text{Ni}_{41.2}\text{Al}_{43.6}\text{Fe}_{12.7}\text{Cr}_{0.8}\text{Mo}_{1.4}$). Assuming that aged and cooling precipitates have a similar contrast, relative to the matrix at the ageing temperatures of 700°C and 800°C, the volume fractions of cooling precipitates at both ageing temperatures can be estimated, based on the USAXS results, and the values are listed in Table 2. Note that quantitative TEM work could be helpful by comparing with the USAXS results.

Figure 2(a) presents the hardness of specimens subjected to different heat treatments. The hardness remains almost constant at $\sim 457 \text{ HV}$ for specimens aged at 800°C, while it slightly decreases at the beginning and stays at $\sim 350 \text{ HV}$ for specimens aged at 700°C. The hardness of specimens aged at 800°C is $\sim 90 \text{ HV}$ higher than that at 700°C. The fact agrees with the observed yield stresses at room temperature ($1,015 \pm 62 \text{ MPa}$ for the specimen aged for 100 h at 700°C + AC (Condition 1); $1,166 \pm 46 \text{ MPa}$ for the specimen aged for 100 h at 800°C + WQ (Condition 2)). The solution-treated specimen has the highest hardness (474 HV). Note that the hardness is associated with the cooling rate, following the ageing treatment at 800°C. The air-cooled specimen is $\sim 27 \text{ HV}$ softer than the water-quenched one. On the contrary, specimens aged at 700°C do not show such strong dependence.

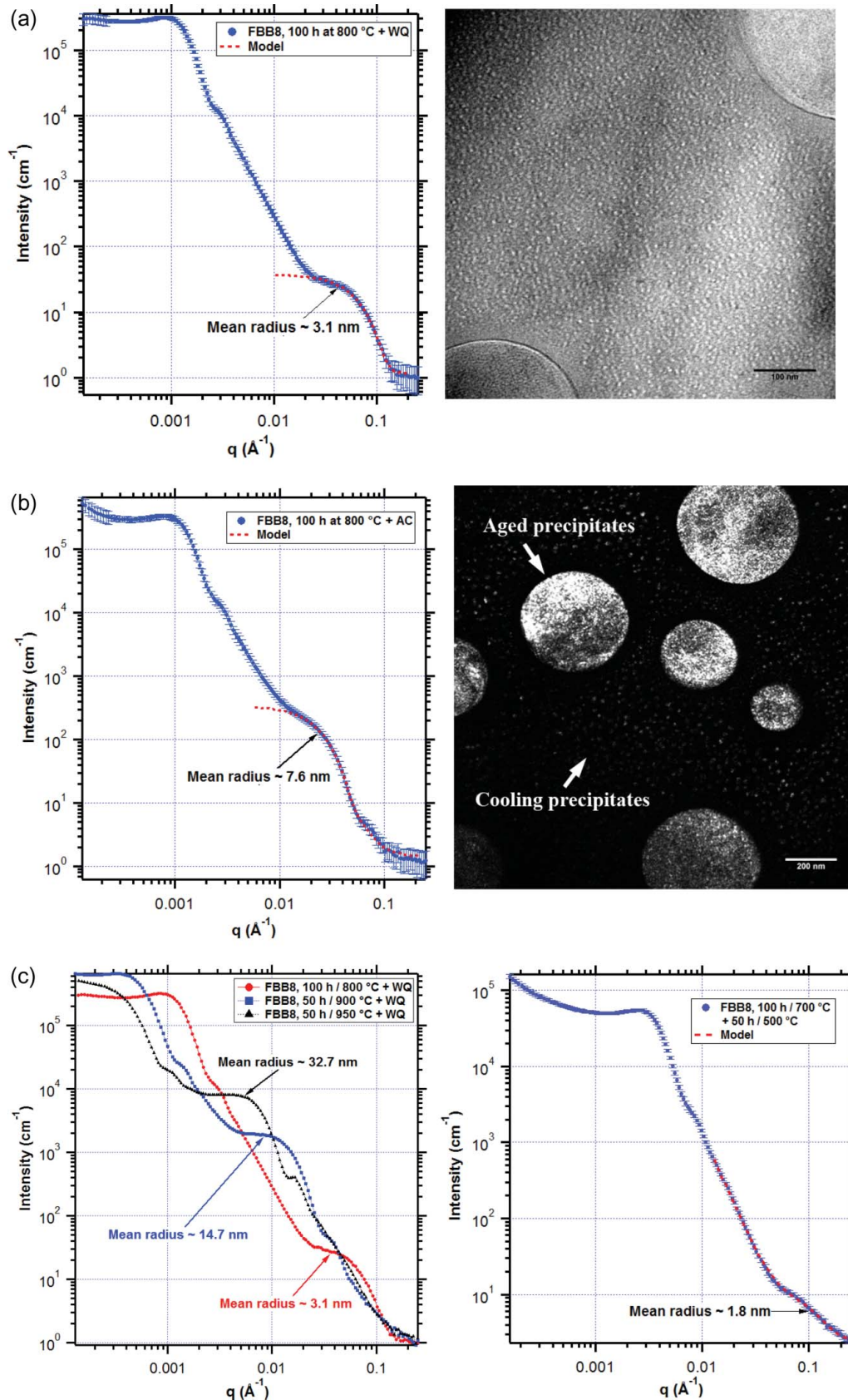


Figure 1. Cooling precipitates in FBB8 aged at 800°C for 100 h followed by WQ (a) and AC (b) characterized by USAXS and TEM. (c) The comparison of cooling precipitates for specimens aged at 800°C, 900°C, and 950°C. (d) Fine precipitates for the specimen aged for 100 h at 700°C + 50 h at 500°C. The dark-field image was taken using (100) reflection from the B2 ordered structure. For clarity, the experimental error bar is not included in (c).

Table 2. Aged and cooling precipitates for Conditions 1 and 2, and their hardening effects.

Specimens	σ_Y (MPa)	Aged precipitates			Cooling precipitates		
		f_1 (%)	$\bar{r}_1$ (nm)	$\Delta\sigma_{Or}$ (MPa)	f_2 (%)	$\bar{r}_2$ (nm)	$\Delta\sigma_{order}$ (MPa)
Condition 1	$1,015 \pm 62$	15.5 ± 0.5 [14]	54 [4]	491 ± 14	~ 3.5	1.5 [14]	~ 322 (Equation 3)
Condition 2	$1,166 \pm 46$	14.6 ± 0.9 [4]	166 [4]	181 ± 10	~ 5.0	3.1 ± 0.5	~ 639 (Equation 4)

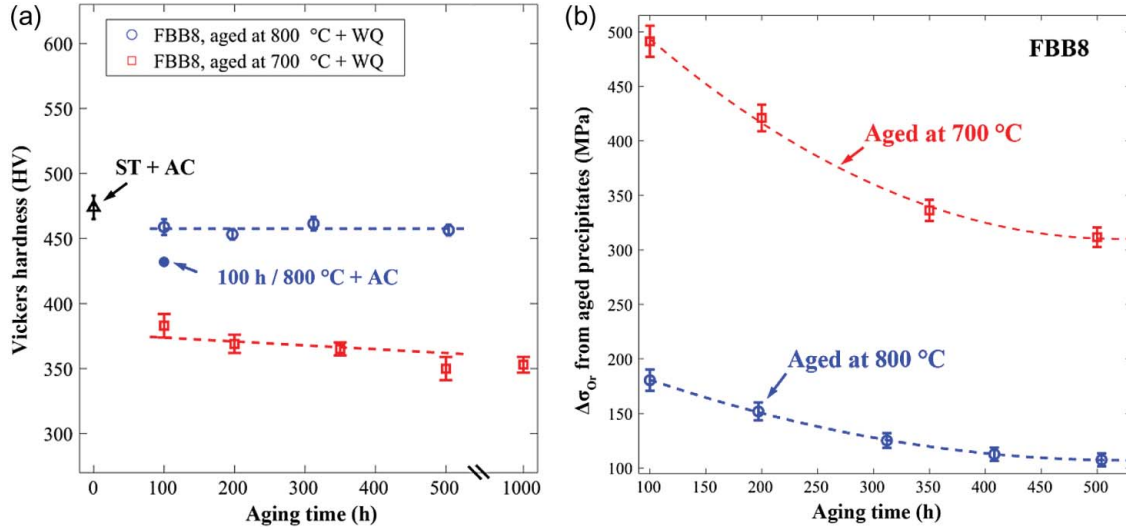


Figure 2. (a) The hardness of specimens subjected to different heat treatments. (b) The strengthening effect of aged precipitates as a function of ageing time.

The TEM micrographs of deformed specimens (Conditions 1 and 2) are shown in Figure 3. In Figure 3(a), dislocation loops (labelled as A) around aged precipitates are observed, indicating that dislocations bypass aged precipitates through the mechanism of Orowan bowing. Meanwhile, dislocations tend to form pairs (labelled as B) in the deformation process. It suggests that dislocations probably cut through fine ordered cooling precipitates.[19] The interaction between dislocations and cooling precipitates in Condition 2 is stronger than that in Condition 1, as shown in Figure 3.

By considering the mean planar inter-particle distance, dislocation dipole effect, and line tension of dislocations, Martin [20] proposed the strengthening effect by Orowan bowing, as described:

$$\Delta\sigma_{Or} = 0.81M \frac{Gb}{2\pi(1-\nu)^{1/2}} \frac{\ln(2r_s/r_0)}{(\lambda_s - 2r_s)}, \quad (1)$$

where G is the shear modulus, b is the length of Burgers vector, ν is the Poisson's ratio, r_s is the mean precipitate radius of intersections, r_0 is the inner cutting-off radius of a dislocation, λ_s is the average planar centre-to-centre spacing between a precipitate and its nearest neighbour, M is the Taylor factor (~ 3), and the factor of 0.81 is added by considering the effective precipitate spacing for a random array of obstacles.[20,21] Geometrically, we

have the following relationships:

$$\lambda_s = \bar{r}_1 \left(\frac{2\pi}{3f_1} \right)^{1/2} \quad \text{and} \quad r_s = \sqrt{2/3} \bar{r}_1. \quad (2)$$

In this study, we take $G = 80$ GPa, $\nu = 0.3$, $b = 0.24982$ nm, and $r_0 = b$. The precipitate volume fractions are 0.155 ± 0.5 [14] and 0.146 ± 0.9 [4] for specimens aged at 700°C and 800°C, respectively. The coarsening behaviour of aged precipitates as a function of the ageing time at 700°C and 800°C was reported by Sun et al. [4]. Based on all these parameters, the hardening contribution from aged precipitates, $\Delta\sigma_{Or}$, is estimated, and the values are plotted against the ageing time at 700°C and 800°C (Figure 2(b)). As expected, the strengthening effect gradually decreases, as the ageing time increases at both temperatures. For example, $\Delta\sigma_{Or}$ goes down from 491 MPa for 100 h to 312 MPa for 500 h at 700°C. Note that $\Delta\sigma_{Or}$ is much high (~ 311 –204 MPa) for specimens aged at 700°C than those aged at 800°C, given the same ageing time.

Dislocations probably cut through fine cooling precipitates, as suggested by Figure 3. Because of the small lattice and modulus mismatch between the matrix and precipitates, the order hardening could dominate the overall strengthening effect from cooling precipitates in this case.[22] The order-strengthening effect is given

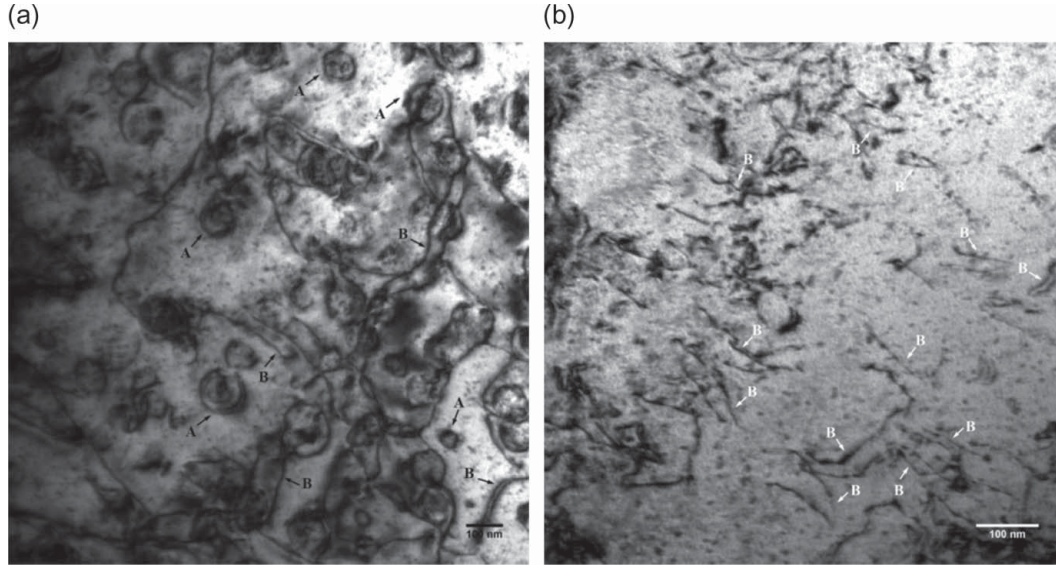


Figure 3. Bright-field images of dislocation structures (A: dislocation loops; B: dislocation pairs) for specimens (a) Condition 1, $\varepsilon \sim -1.7\%$; (b) Condition 2, $\varepsilon \sim -0.55\%$.

by [22]

$$\Delta\sigma_{\text{order}} = M \frac{\gamma_{\text{apb}}}{2b} \left(\frac{3\pi^2 \gamma_{\text{apb}} f_2^* \bar{r}_2}{32\Gamma} \right)^{1/2}, \quad (3)$$

where γ_{apb} ($\sim 0.5 \text{ J/m}^2$ [7,23]) is the anti-phase boundary energy on the (110) plane of NiAl-type precipitates, and Γ is the dislocation line tension ($\sim 1/2 Gb^2$). The effective volume fraction of f_2^* , defined as $f_2^* = f_2/(1 - f_1)$, is used instead of f_2 , as suggested by Repich et al.[24] When precipitates are strong enough (the breaking angle of dislocations $< \sim 120^\circ\text{C}$ [22]), the order-strengthening effect becomes [22]

$$\Delta\sigma_{\text{order}} = 0.81M \frac{\gamma_{\text{apb}}}{2b} \left(\frac{3\pi f_2^*}{8} \right)^{1/2}. \quad (4)$$

Based on the TEM micrograph (Figure 3(b)), Equation (4) is probably more suitable to calculate the strengthening effect of cooling precipitates in Condition 2. The estimation of $\Delta\sigma_{\text{order}}$ of cooling precipitates in Conditions 1 and 2 is listed in Table 2, based on the microstructural information. $\Delta\sigma_{\text{order}}$ is $\sim 322 \text{ MPa}$ for Condition 1, while it is approximately 2.5 times larger than $\Delta\sigma_{\text{Or}}$ in the case of Condition 2. We conclude that cooling precipitates harden the matrix considerably at room temperature in both conditions, besides the strengthening effect provided by aged precipitates.

The fractured specimens after tensile tests were examined by SEM. The specimen shows the river pattern of the cleavage transgranular fracture and cracked NiAl-type precipitates (labelled as C) just below the fracture surface are observed (Figure 4). NiAl is brittle at room temperature in both single-crystalline and polycrystalline forms.[25,26] It is very likely that cracks

initiate from NiAl-type precipitates themselves. Cooling precipitates could have two impacts on the room-temperature fracture. First, they excessively harden the matrix, and, hence, the yield stress may exceed the fracture stress of NiAl-type precipitates. It is worth noticing that the stress is probably inhomogeneous on the microscopic scale during deformation.[20] Due to the excessive hardening of cooling precipitates, the plastic zone size ($\propto 1/\sigma_Y^2$) near the crack tip is expected to be small, which results in a small amount of energy absorption by plastic deformation.[27] Hence, once a crack forms, it could propagate quickly through the matrix. The coalescence and growth of cracks (labelled as P) are also observed in Figure 4. Teng et al. [11] studied the effect of the ageing time (up to 1,000 h at 700°C) on the room-temperature ductility of FBB8. They concluded that the ductility is independent of the ageing time, or equally the precipitate size in the studied range. $\bar{r}_1$ becomes 122 nm for the FBB8 specimen aged for 1,000 h at 700°C .[4] and the corresponding $\Delta\sigma_{\text{Or}}$ decreases to $\sim 250 \text{ MPa}$. However, the hardness of the specimen stays invariantly at $\sim 350 \text{ HV}$ (Figure 2(a)) due to the strengthening effect of cooling precipitates. It is possibly why the room-temperature ductility of FBB8 does not improve even after a long-time ageing treatment in their study.[11] New composition designs by controlling the amounts of aluminium and nickel and their ratio in order to decrease the solubility of aluminium or nickel in the matrix at the ageing temperature are perhaps ways to improve the ductility of NiAl-strengthened ferritic alloys at room temperature. The refinement of grain size by the hot deformation and following heat treatment may also help.

In conclusion, duplex precipitates in FBB8 were observed by USAXS and TEM. Fine precipitates

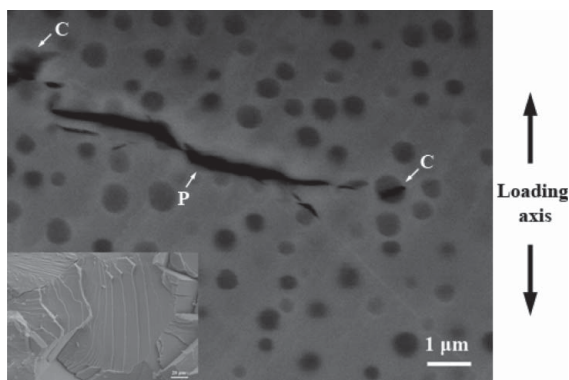


Figure 4. The cracked precipitates just below the fracture surface (C: cracked precipitates; P: coalescence and growth of cracks). The specimen was aged for 207 h at 800°C. Typical fractography is also shown in the inset.

nucleate and grow in the cooling process, following the ageing treatment. The average radius of these cooling precipitates ranges from several to tens nanometres, depending on the cooling rate and ageing temperature. At room temperature, dislocations bypass aged precipitates through the mechanism of Orowan bowing, while fine cooling precipitates are possibly cut through by dislocations. Cooling precipitates harden the matrix considerably at room temperatures, and the order-strengthening effects from cooling precipitates are estimated to be 322 and 639 MPa for Conditions 1 and 2, respectively. For the room-temperature fracture of FBB8, cracks probably initiate from NiAl-type precipitates, and they could propagate quickly through the matrix due to the excessive hardening contribution from cooling precipitates.

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