INVESTIGATION ON THE MICROSTRUCTURE CHANGES OF NiCrAlY COATING DUE TO SHOT PEENING PLASTIC DEFORMATION AND AGING

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ABSTRACT

NiCrAlY coating achieved by means of arc plasma deposition (APD) was used to protect DD3, a kind of nickel base single crystal superalloy, from elevated temperature oxidation. Phase analysis, microstructure and density of the coating in different states including diffused, plastic deformed and aged after peening plastic deformation have been investigated. It has been showed that the shot peening plastic deformation can not only transform order Ni_3Al phase, matrix of coating, into disorder one as well as dissolve NiAl phase precipitated during diffusion treatment but also increase the density by 30%. It has also been pointed out that NiAl phase can be precipitated again from plastic deformed matrix Ni_3Al at 1100 ℃. The coating can be strengthened by the NiAl phase precipitation during aging treatment. Both increase of density and precipitation of NiAl phase during aging treatment result in the improvement of resistance to the elevated temperature oxidation for NiCrAlY coating.

Key word

Shot peening, coating resistance to oxidation, plastic deformation, phase transformation, density

1   INTRODUCTION

The elevated temperature oxidation resistance of nickel based superalloys can be effectively increased by thermal barrier coating. In order to achieve better oxidation resistance and excellent combination between matrix and ceramic coating, a bond coating must be used. NiCrAlY coating , 130 μm in thickness, which is used in the present experiment is a kind of bond coating achieved by means of arc plasma deposition(APD) used to protect DD3, a kind of nickel base single crystal materials, from elevated temperature oxidation in the present work. Three states of coating have been investigated.

1. Original coating(OC): arc plasma deposition.
2. Diffusion coating(DC): arc plasma deposition+870℃(1h) (diffusion treatment)
3. Shot peening coating(SPC): DC+ shot peening
The changes of phases transformation, microstructure and density of the various coatings can have certain impact on the elevated temperature oxidation resistance.

2 MATERIAL AND EXPERIMENTAL PROCEDURE

2.1 Material

The matrix is DD3, a kind of nickel base single crystal superalloy, and the coating is NiCrAlY. Their main chemical compositions are listed in Table 1 and Table 2 respectively.

TABLE 1　Main chemical compositions of DD3 nickel base single crystal superalloy (wt%)

Cr	Co	W	Mo	Ta	Ti	Ni
~8	~10	~6	~0.5	~6	~1	remain

TABLE 2　Chemical composition of NiCrAlY coating (wt%)

Ni	Cr	Al	Y
66.9~69.9	22~23	8~10	0.1~0.3

2.2　Experimental procedure

The microstructure and phase analysis by X-ray were carried on the Rigaku X-Ray Diffractometer of D/max~2400 and D9C. The density was tested on balance with 10-4g sensitivity. The thickness of coating were measured with ±1 μm sensitivity. The size of specimen is 1 × 20 × 30mm.

Procedures of elevated temperature oxidation: The specimen were put into furnace for 55 minutes at 1150℃ and took out for 5 minutes cooling in the air, which is named one cycle(1h). There were only 4 cycles(4h) for DD3 measuring weight every hour. There were 70 cycles for DC(70h) and 90 cycles for SPC(90h) measuring weight by the hour of 1, 4, 7, 10, 20, 30, 40, 50, 60, 70, 80, 90.

3 RESULT AND DISCUSSION

3.1 The phase structure of DC and SPC

The microstructure of OC and its distribution along the depth has been reported[1]. The microstructure of DC along the depth was presented in Table 3. It shows that order Ni_3Al and Cr phase precipitated along the whole depth of coating, which is about 130 μm . The NiAl, a simple cubic crystal, precipitated only within the depth of 45 μm which does not exist in OC.

The microstructure of SPC within 10 μm has been shown in Table 4. The phase transformation and microstructure can be changed greatly due to cyclic plastic deformation induced by shot peening. First, NiAl precipitated during diffusion treatment is dissolved into the matrix Ni_3Al. Second, plastic deformation can transform order Ni_3Al phase into disorder one but has no influence on the morphology and distribution of Cr phase.

TABLE 3 The phase structures of DC at different depths

DC d	DC (hkl)	DC—16 µm d	DC—16 µm (hkl)	DC—33 µm d	DC—33 µm (hkl)	DC—44 µm d	DC—44 µm (hkl)	DC—67 µm d	DC—67 µm (hkl)
3.5871	Ni_3Al (100)	3.5871	Ni_3Al (100)	3.5814	Ni_3Al (100)	3.5786	Ni_3Al (100)	3.5871	Ni_3Al (100)
2.8988	**NiAl (100)**	2.8806	**NiAl (100)**	2.8842	**NiAl (100)**	2.5294	Ni_3Al (110)	2.5322	Ni_3Al (110)
2.5335	Ni_3Al (110)	2.5349	Ni_3Al (110)	2.5322	Ni_3Al (110)	2.0652	Ni_3Al (111)	2.0670	Ni_3Al (111)
2.0679	Ni_3Al (111)	2.0688	Ni_3Al (111)	2.0661	Ni_3Al (111)	2.0386	Cr (110) / **NiAl (110)**	2.0413	Cr (110)
2.0413	Cr (110) / **NiAl (110)**	2.0421	Cr (110) / **NiAl (110)**	2.0404	Cr (110) / **NiAl (110)**	1.7873	Ni_3Al (200)	1.7892	Ni_3Al (200)
1.7873	Ni_3Al (200)	1.7905	Ni_3Al (200)	1.7886	Ni_3Al (200)	1.5989	Ni_3Al (210)	1.5999	Ni_3Al (210)
1.6004	Ni_3Al (210)	1.6009	Ni_3Al (210)	1.5999	Ni_3Al (210)	1.4593	Ni_3Al (211)	1.4605	Ni_3Al (211)
1.4609	Ni_3Al (211)	1.4613	Ni_3Al (211)	1.4601	Ni_3Al (211)	1.4411	Cr (200) / **NiAl (200)**	1.4419	Cr (200)
1.4419	Cr (200) / **NiAl (200)**	1.4427	Cr (200)	1.4419	Cr (200) / **NiAl (200)**	1.2639	Ni_3Al (220)	1.2645	Ni_3Al (220)
1.2646	Ni_3Al (220)	1.2650	Ni_3Al (220)	1.2645	Ni_3Al (220)	1.1765	Cr (211) / **NiAl (211)**	1.1769	Cr (211)
1.1769	Cr (211) / **NiAl (211)**	1.1772	Cr (211) / **NiAl (211)**	1.1767	Cr (211) / **NiAl (211)**	1.0777	Ni_3Al (311)	1.0781	Ni_3Al (311)
1.0783	Ni_3Al (311)	1.0785	Ni_3Al (311)	1.0779	Ni_3Al (311)	1.0320	Ni_3Al (222)	1.0321	Ni_3Al (222)
1.0325	Ni_3Al (222)	1.0326	Ni_3Al (222)	1.0321	Ni_3Al (222)	1.0188	Cr (220)	1.0191	Cr (220)
1.0188	Cr (220)	1.0191	Cr (220)	1.0192	Cr (220)	0.9556	**NiAl (300)**	0.9114	Cr (310)
0.9129	Cr (310)	0.9558	**NiAl (300)**	0.9558	**NiAl (300)**	0.9112	Cr (310)	0.8937	Ni_3Al (400)
0.8938	Ni_3Al (400)	0.9115	Cr (310)	0.9116	Cr (310)	0.8932	Ni_3Al (400)	0.8321	Cr (222)
0.8318	Cr (222)	0.8939	Ni_3Al (400)	0.8935	Ni_3Al (400)	0.8321	Cr (222)		
		0.8318	Cr (222)	0.8318	Cr (222)				
		0.8203	Ni_3Al (331)						

TABLE 4 The phase structures of SPC in surface layer

| No	Measurement result d (A)	Measurement result I	Analyzing results Ni_3Al d (A)	Analyzing results Ni_3Al I	Analyzing results Ni_3Al (hkl)	Analyzing results Cr d (A)	Analyzing results Cr I	Analyzing results Cr (hkl)
1	2.077	100	2.077	100	(111)			
2	2.043	67				2.039	100	(110)
3	1.809	24	1.799	70	(200)			
4	1.444	10				1.441	16	(200)
5	1.272	43	1.265	60	(220)			
6	1.179	23				1.177	30	(211)
7	1.084	1	1.078	60	(311)			
8	1.037	3	1.032	40	(222)			
9	1.0216	4				1.019	18	(220)
10	0.9136	5				0.912	20	(310)
11	0.8340	12				0.832	6	(222)
12	0.8245	8	0.819	70	(331)			

3.2 The microstructure changes of coating due to plastic deformation

The changes of the diffraction angle 2θ, the integral breadth B and the background diffusion intensity I_b near the Bragg angle for (200) diffraction line with the shot peening intensities (represented by air pressure, P) within plastic deformed layer have been tested (Fig. 1). The higher the shot peening intensity, the greater the plastic deformation extent in the surface layer. The changes of the B—P curves shows that the lattice distortion ($\Delta a/a$, a--lattice parameter) of

Ni$_3$Al becomes larger and the size of the subgrain becomes smaller with the increase of plastic deformation amount. The changes of the I$_b$—P curves shows that the value of I$_b$ increases from 3000 impulse counts per second (c/s) to 3500 c/s because of plastic deformation but no obvious changes on I$_b$ have been found with the extent of deformation. That means the value of I$_b$ can be reached its saturation (3500 c/s) by only slight deformation (P=0.1 MPa). The more intensity I$_b$ means that the amount of mosaic in the unit volume   participated   in   non-coherent   diffusion increases. On the other hand,   the density of coating is raised. The changing trend of the 2θ — P curves indicates that 2θ gets smaller with the increase of shot peening intensity, which means the introduction of residual stresses into the surface layer due to cyclic plastic deformation caused by shot peening. It will be published in another paper about residual stresses and its distribution due to shot peening within coating.
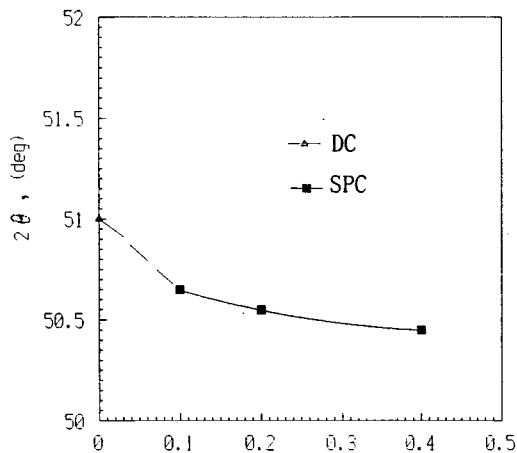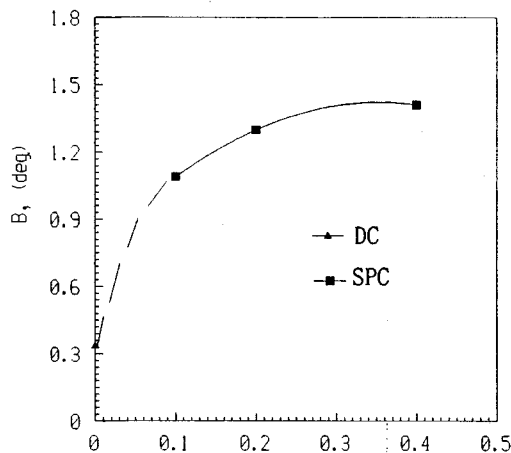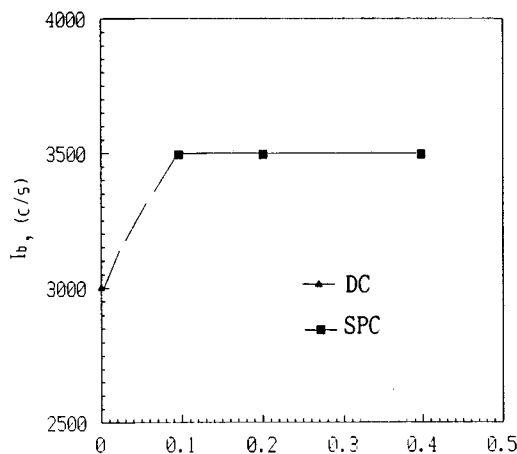
Fig 1. The relationships of diffraction angle 2θ of (200) line (a), the integral breadth B of (200) diffraction line (b) and diffusion intensity I$_b$ near the Bragg angle of (200) line (c) with the shot peening intensities(represented by air pressure, MPa)   within plastic deformed layer

3.3 The influence of aging on the phase structure and microstructure of SPC

The changes of the phase structure during aging have been shown in Table 5. It can be seen that disorder Ni$_3$Al can be transform into order one entirely during the process of aging. The main two phases are order Ni$_3$Al and Cr after aging at 500°C~1000°C. The simple-cubic NiAl would precipitate when the temperature goes up to 1100°C(listed in the last column of Table 5).

TABLE 5 The changes of phase structures of SPC with aging temperature

peening		peening+500°C		Peening+600°C		peening+800°C		peening+1000°C		peening+1100°C	
d	(hkl)	d	(hkl)	D	(hkl)	d	(hkl)	d	(hkl)	d	(hkl)
2.0742	Ni$_3$Al (111)	3.5814	Ni$_3$Al (100)	3.5814	Ni$_3$Al (100)	3.5786	Ni$_3$Al (100)	3.5786	Ni$_3$Al (100)	3.5786	Ni$_3$Al (100)
2.0456	Cr (110)	2.5294	Ni$_3$Al (110)	2.5280	Ni$_3$Al (110)	2.5280	Ni$_3$Al (110)	2.5294	Ni$_3$Al (110)	2.8697	NiAl (100)
1.8024	Ni$_3$Al (200)	2.0652	Ni$_3$Al (111)	2.0643	Ni$_3$Al (111)	2.0643	Ni$_3$Al (111)	2.0652	Ni$_3$Al (111)	2.5294	Ni$_3$Al (110)
1.4451	Cr (200)	2.0404	Cr (110)	2.0413	Cr (110)	2.0413	Cr (110)	2.0395	Cr (110)	2.0652	Ni$_3$Al (111)
1.2688	Ni$_3$Al (220)	1.7873	Ni$_3$Al (200)	1.7873	Ni$_3$Al (200)	1.7873	Ni$_3$Al (200)	1.7879	Ni$_3$Al (200)	2.0386	Cr (110) NiAl (110)
1.1800	Cr (211)	1.5979	Ni$_3$Al (210)	1.5984	Ni$_3$Al (210)	1.5984	Ni$_3$Al (210)	1.5989	Ni$_3$Al (210)	1.7873	Ni$_3$Al (200)
1.0844	Ni$_3$Al (311)	1.4585	Ni$_3$Al (211)	1.4585	Ni$_3$Al (211)	1.4589	Ni$_3$Al (211)	1.4597	Ni$_3$Al (211)	1.6565	NiAl (111)
1.0359	Ni$_3$Al (222)	1.4427	Cr (200)	1.4427	Cr (200)	1.4427	Cr (200)	1.4415	Cr (200)	1.5989	Ni$_3$Al (210)
1.0219	Cr (220)	1.2636	Ni$_3$Al (220)	1.2630	Ni$_3$Al (220)	1.2630	Ni$_3$Al (220)	1.2639	Ni$_3$Al (220)	1.4597	Ni$_3$Al (211)
0.9134	Cr (310)	1.1779	Cr (211)	1.1772	Cr (211)	1.1897		1.1767	Cr (211)	1.4415	Cr (200)
0.8342	Cr (222)	1.0774	Ni$_3$Al (311)	1.0768	Ni$_3$Al (311)	1.1774	Cr (211)	1.0777	Ni$_3$Al (311)	1.2636	Ni$_3$Al (220)
0.8227	Ni$_3$Al (331)	1.0309	Ni$_3$Al (222)	1.0310	Ni$_3$Al (222)	1.0770	Ni$_3$Al (311)	1.0318	Ni$_3$Al (222)	1.1765	Cr (211)
		1.0192	Cr (220)	1.0191	Cr (220)	1.0310	Ni$_3$Al (222)	1.0189	Cr (220)	1.1708	NiAl (211)
		0.9125	Cr (310)	0.9120	Cr (310)	1.0194	Cr (220)	0.9112	Cr (310)	1.0774	Ni$_3$Al (311)
		0.8932	Ni$_3$Al (400)	0.8922	Ni$_3$Al (400)	0.9123	Cr (310)	0.8938	Ni$_3$Al (400)	1.0318	Ni$_3$Al (222)
		0.8322	Cr (222)	0.8321	Cr (222)	0.8923	Ni$_3$Al (400)	0.8315	Cr (222)	1.0189	Cr (220)
		0.8200	Ni$_3$Al (331)	0.8199	Ni$_3$Al (331)	0.8323	Cr (222)	0.8200	Ni$_3$Al (331)	0.9550	NiAl (300)
						0.8197	Ni$_3$Al (331)			0.9154	Cr (310)
										0.9108	NiAl (310)
										0.8932	Ni$_3$Al (400)
										0.8318	Cr (222)
										0.8198	Ni$_3$Al (331)

Fig 2. shows the change of the integral breadth B of (200) diffraction line and the diffusion intensity I_b near the Bragg angle of (200) line with aging temperature in the plastic deformed layer. Fig 3 shows the changes of the integral breadth B of (211) diffraction line for the overlapping of both Cr and NiAl and diffusion intensity I_b near the Bragg angle for (211) line with aging temperature. The changes of both parameters, B and I_b, reflect the changes of phases structure during aging.

It can be seen from I_b in Fig 2,b and Fig 3,b that I_b of DC is the lowest and I_b of SPC is higher than that of DC. The I_b for any kind of coating is not influenced by aging treatment. This results are consistent with that achieved from Ni$_3$Al in Fig 1,c, which indicates that the density of coating can be increased by cyclic deformation due to shot peening.

The curves of B—T in Fig 2,a and Fig 3,a indicates the changes of microstructure of matrix Ni$_3$Al and NiAl phase. The gradual decline of the values of B for plastic deformed Ni$_3$Al within 600 °C shows that there is occurrence of recovery for deformed microstructure of Ni$_3$Al. However, the values of B drop down sharply after 600°C and remain steady, which is basically consistent to that of Ni$_3$Al for DC, within the temperature range of 800°C~1000°C. The results indicate that recrystallization begin for plastic deformed Ni$_3$Al at 600 °C and end at 800 °C, but no recrystallization coagulation appear even at 1000 °C. The values of B can be increased again when temperature goes up to 1100 °C. The reason for the phenomenon is that NiAl can be precipitated resulting in dispersed strengthening to the matrix Ni$_3$Al at 1100 °C(shown in the last column of Table 5). It can be also seen from Fig 3,a that B of (211) is contributed only by Cr

before the precipitation of NiAl under 1000°C. When the aging temperature is reached to 1100°C，NiAl can be precipitated. In such case, the diffraction lines of (211) for Cr and NiAl are overlapping because diffraction angles for both phases are equal, $2\theta = 81.72°$. Just for the precipitation of NiAl at 1100°C, B is increased again(Fig 3,a). The precipitation of NiAl enables the lattice of Ni₃Al to distort leading to dispersed strengthening of matrix. This is the reason of suddenly widening again of B of matrix aging at 1100°C(Fig 2,a). After aging treatment at 1100 °C, the elevated temperature strength as well as elevated temperature resistance to the oxidation of SPC can certainly be improved due to the precipitation of NiAl phase and dispersed strengthening of matrix Ni₃Al.
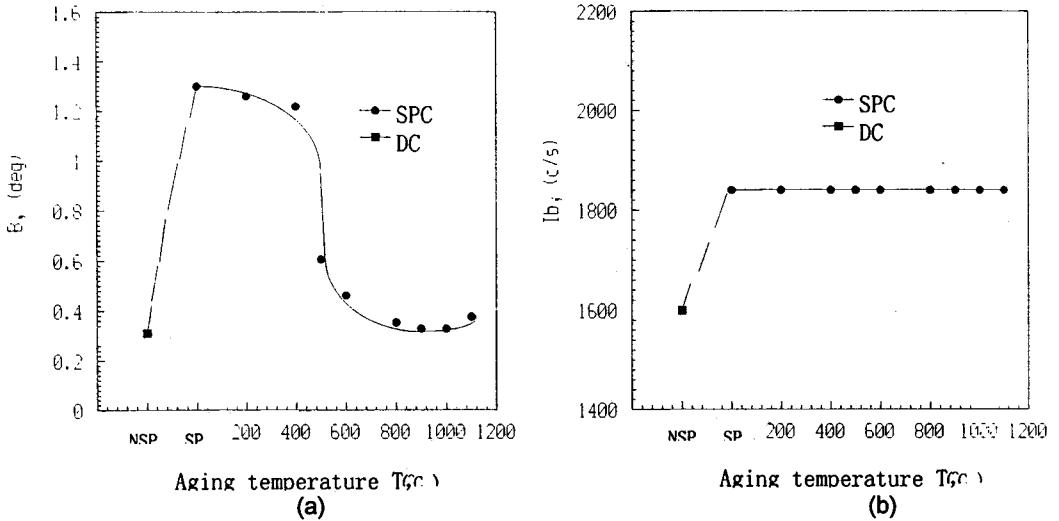

Fig 2. The changes of the integral breadth B of (200) diffraction line (a) and diffusion   intensity Ib near the Bragg angle of (200) line (b) for Ni₃Al with aging temperature in the plastic deformed layer
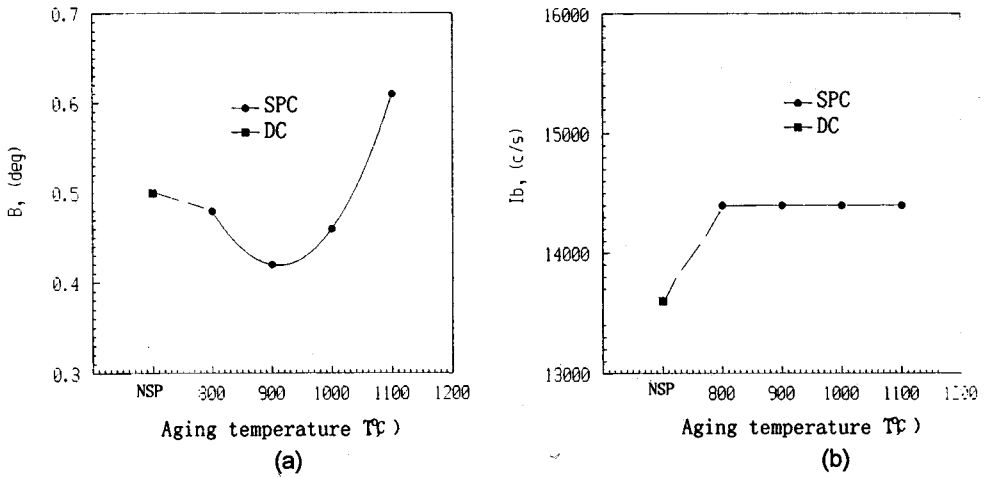

Fig 3. The changes of the integral breadth B of (211) diffraction line for the overlapping of both Cr and NiAl(a) and diffusion intensity Iᵦ near the Bragg angle of (211) line (b) with aging temperature

3.4 The influence of plastic deformation on the density of coating

The results from Fig 2,b and Fig 3,b show the density of coating can be increased due to shot peening induced plastic deformation. The measurement on density were carried on for DC and SPC and the results have been listed in Table 6. The density fluctuations for DC is ρ_{DC} =4.439~6.341g/cm³ because of technology unsteady of APD. The scattering range is about ± 0.95g/cm³. But the scattering range of density can be greatly decreased by shot peening. Its value is ρ_{SPC} =7.057g/cm³ which is very near to the theoretical value, ρ_T =7.076g/cm³, calculated from compositions in Table 2. It can be judged from I_b—P in Fig 2,b and Fig 3,b that the magnitude of shot peening intensity has no obvious impact on the density which means that the density can be increased by nearly 30% even at lower peening intensity. There is no evident influence on density by increasing intensity but can greatly improve the microstructure of coating.

It is clearly that the improvement of density meaning the reduction of microporosities and other metallurgical defects can effectively increase the resistance to the diffusion of oxygen element into the coating, resulting in the improvement of resistance to the oxidation of coating.

TABLE 6 The results of density measurement for diffusion coating before and after shot peening

thickness of coating (mm)		weight of coating (g)		density of DC(ρ_{DC}) (g/cm³)	density of SPC(ρ_{SPC}) (g/cm³)	increase of density (%)
before peening	after peening	before peening	after peening			
0.3492	0.3080	1.3284	1.3135	6.341	7.108	12.1
0.3090	0.2250	1.0161	0.9565	5.480	7.085	29.3
0.3860	0.2440	1.0289	1.0219	4.439	6.980	57.2

note: (1)The average density for three measurements: ρ_{DC} =5.420g/cm³, ρ_{SPC} =7.057g/cm³;
(2)The average density increase by shot peening: 30%
(3)The theoretical value of density calculated from Table 2:

$$\rho_T = 1/(\frac{0.683}{8.9} + \frac{0.225}{7.2} + \frac{0.09}{2.7}) = 7.076 \text{ g}/cm^3$$

3.5 The influence on the resistance of elevated temperature oxidation due to shot peening

The test results of elevated temperature oxidation for three kind of specimens, DD3 without coating, DC and SPC, have been shown in Fig. 4. The data of Table 7 can be achieved by analyzing the curves of weight gain versus time(G-T) in Fig. 4. It can be seen from Fig. 4 and Table 7 that:

(1)The DD3 superalloy without coating does not resist the elevated temperature oxidation.

(2)The resistance to oxidation for DC can be further increased by shot peening which means that SPC has the best property of resistance to elevated temperature oxidation.

Considering the balance cycles for different coatings at which the weight gain is equal to weight lose(Δ G=0), it is clear that the life DC can be increased 42% by shot peening strengthening(SPC).

TABLE 7 Comparison of the oxidation properties between DC and SPC

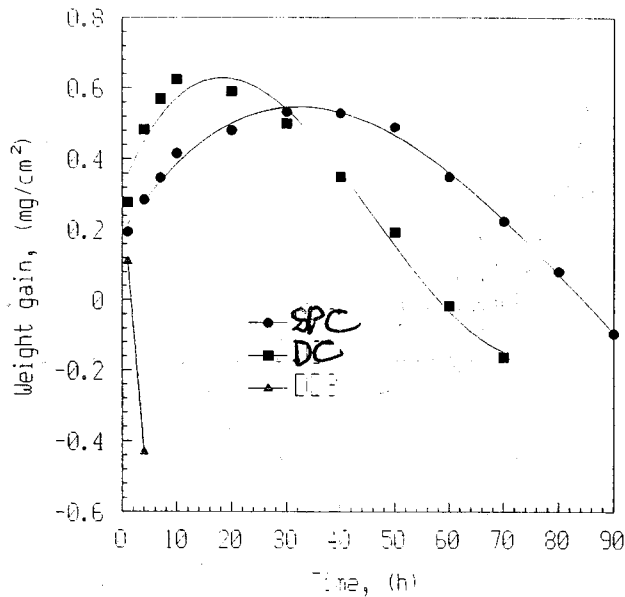| Tested parameters	coating		relative change of parameters, (%)
	DC	SPC	
Maximum weight gain, ΔG (mg/cm²)	0.63	0.52	-17.5
The cycles for Maximum weight gain (cycle)	10	40	+300
Balance cycles(Δ G=0), (cycle)	~60	~85	+42

Fig. 4 Weight changes(G) versus time(T)of DD3 without coating, DC and SPC specimens during elevated temperature oxidation experiment

To sum up all factors mentioned above, the reasons of SPC with higher resistance to elevated temperature oxidation than DC are:

(1) The density of DC can be increased by 30% due to shot peening(Table 6). The improvement of density certainly promotes the reduction of microporosities and other metallurgical defects resulting in the decrease of diffusion speed of oxygen element from the surface into the coating.

(2)The precipitation of NiAl from matrix Ni_3Al during aging at 1100℃ leads to dispersed strengthening, which improves the resistance to the elevated temperature oxidation (Fig 3,a).

4 CONCLUSION

(1)The changes of phase structure and microstructure of DC due to cyclic plastic deformation are: order Ni_3Al transforms into disorder Ni_3Al; NiAl precipitated in DC can be dissolved again into the matrix; the lattice distortion of matrix and Cr phase takes place.

(2)There is occurrence of recovery for deformed microstructure of Ni_3Al below 600℃ during aging process, then recrystallization begins at 600℃. The precipitation of NiAl takes place at 1100 ℃.

(3)The average density of DC can be increased by 30% due to shot peening, which is approximately equal to the theoretical value, ρ_T =7.076g/cm³.

(4)Both increment of density and precipitation of NiAl phase during aging treatment for shot peening coating NiCrAlY result in the improvement of resistance to the elevated temperature oxidization.

(5)The new application area has been found by the shot peening technology in the improvement of microstructures and physical properties of coatings.

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