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Effect of heat treatment on microstructure and tensile strength of NiCoCrAl alloy sheet fabricated by EB-PVD

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Abstract: The NiCoCrAl alloy sheet was fabricated by electron beam physical vapor deposition technique and the effects of the heat treatment on the microstructure and tensile strength of the NiCoCrAl alloy sheet were investigated. The heat treatment at 1050 °C is favorable to improve the interface bonding between the columnar structures due to the disappearance of the intergranular gaps. Comparing with the thin NiCoCrAl alloy sheet before heat treatment, the Ni_3Al phase appears in the NiCoCrAl alloy sheet after heat treatment, which is favorable to improve the interface bonding between the columnar structures. The increase in the tensile strength and elongation is attributed to the improvement of the interface bonding between the columnar structures. The residual stress in the NiCoCrAl alloy sheet after heat treatment is reduced significantly, which also confirms that the interface bonding is improved by the heat treatment.

Key words: nickel-base alloys; heat treatment; microstructure; mechanical properties; physical vapor deposition

1 Introduction

Energy saving and environmental preservation are important issues for us to be urgently resolved. Since reducing the weight of vehicles is one of the efficient countermeasures against them, the use of the alloy has been increased in fabricating the vehicles [1]. Furthermore, the alloy sheets have been developed for a variety of applications in high-technology fields, such as the aerospace and automobile industries, as well as in sporting goods [2,3]. A use of high specific strength alloy sheets has increased in the vehicle weight reduction [3]. Especially, the alloy sheets of super thin types are targeted as materials for body panel because they are heat-treatable and have the merits of not only lowering a flow stress by solution treatment, which is advantageous to press forming, but also increasing the strength by baking in the succeeding painting operation [4]. However, the alloy sheets including aluminum are apt to form a gap in press forming compared with other alloy sheets [5]. The combination of properties of the alloy sheets of super thin types was sharply reduced due to the presence of the gap in the alloy sheets. The solution will be grain refinement and various attempts using special manufacturing techniques, such as accumulative roll bonding, differential speed rolling and electron beam physical vapor deposition (EB-PVD), have been made [6−8].

Among the special manufacturing techniques, the electron beam physical vapor deposition (EB-PVD) is a high efficiency and non-equilibrium deposition technique [9]. It is commonly used to deposit the coatings on rotating blades and some high-pressure turbine section vanes. The unique columnar microstructure is typical of thin layer component obtained by EB-PVD, which can provide outstanding resistance against thermal shock and mechanical strains [10]. It was recognized that a grain

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size of the current production of the alloy sheets is determined by their final heat treatments because the long term heating process makes the grain of the alloy sheets fine [11,12].

In the present work, the NiCoCrAl alloy sheet of super thin type was fabricated by EB-PVD technique and the tensile strength of the fabricated NiCoCrAl alloy sheet was determined. Furthermore, the effect of the heat treatment on the microstructure and tensile strength of the NiCoCrAl alloy sheet was investigated in detail.

2 Experimental

During deposition process, the stainless steel substrate with the diameter of 1 m and surface roughness (R_a) of 1.0 rotated at 6 r/min around the vertical axis and the substrate temperature was maintained at (650±5) °C approximately. The process pressure was in the range of $(6-10)\times10^{-3}$ Pa. Ni−20%Co−12%Cr−4%Al ingot with 68 mm in diameter and 250 mm in length was used as the evaporation source. A small amount of CaF_2 was evaporated and a thin (5−10 μm) separating layer of CaF_2 was deposited on the substrate surface. Following deposition, the NiCoCrAl alloy sheet was removed from the substrates. Tensile strength tests based on GB 6397—86 were carried out by an INSTRON−5569 universal materials testing machine with a crosshead displacement speed of 0.05 mm/min at room temperature. The NiCoCrAl alloy sheet was heat treated for different time in Ar atmosphere, and a heating rate of 10 °C/min and a cooling rate of 5 °C/min were lower than the heating cooling rate of the NiCoCrAl alloy sheet in the fabrication process. In this work, five specimens were tested to get an average value. The microstructure observations of specimens were examined by SEM (FEI QUANTA200). The residual stress in the NiCoCrAl alloy sheet before and after the heat treatment at (1050±5) °C was quantified by using X-ray diffractometer (Philips, X'Pert-MRD). The phase composition of the NiCoCrAl alloy sheet was determined by X-ray diffractometer (Philips, X'Pert-MRD).

3 Results and discussion

3.1 Microstructure

Figure 1 shows the surface SEM images of the NiCoCrAl alloy sheet before heat treatment and the uniform particles were observed on the surface of the NiCoCrAl alloy sheet. The large number of obvious humps were the typical deposition phenomena of EB-PVD technique, which was attributed to the priority growth of the deposition particles [13]. The clear boundary located between the humps was readily detected, as shown in Fig. 1(a), and the further observation indicated that the interface bonding between the particles in the hump was closer than the interface bonding located between the humps, as shown Fig. 1(b). Such microstructure was attributed to the columnar growth during the EB-PVD process [14].

The SEM images of the fractured surface of the NiCoCrAl alloy sheet before heat treatment for the tensile strength test are shown in Fig. 2. The fractured surface showed the clear columnar structure and the presence of the gap parallel to the clear columnar structure was due to the effect of the tensile force [13,14]. The obvious plastic deformation was observed in Fig. 2(b), which revealed good interface bonding between columnar structures. Furthermore, the tensile strength of the NiCoCrAl alloy sheet was mainly affected by the interface bonding between columnar structures because the NiCoCrAl alloy sheet was fractured along the interface between columnar structures [14].

It was recognized that the combination of the properties of the alloy sheets could be improved by the heat treatments [11,12]. The SEM images of the fractured surface of the NiCoCrAl alloy sheet heat treated at 950, 1050 and 1150 °C, respectively, for 40 min are shown in Fig. 3. The width of the gap parallel to the columnar structure for the specimen heat treated at 950 °C for 40 min was obviously increased, as shown in

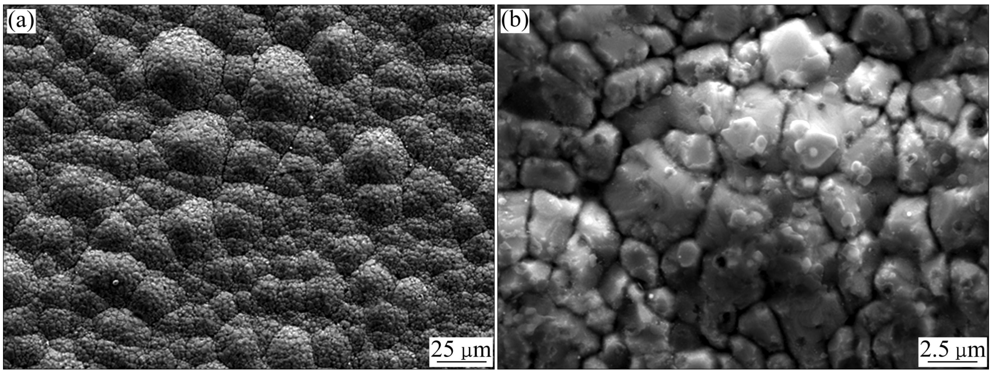

Fig. 1 Surface SEM images of NiCoCrAl alloy sheet

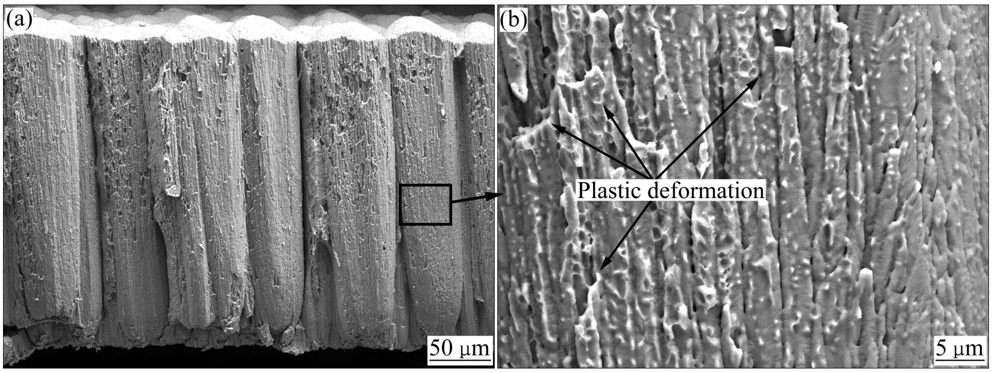

Fig. 2 SEM images of fractured surface of NiCoCrAl alloy sheet

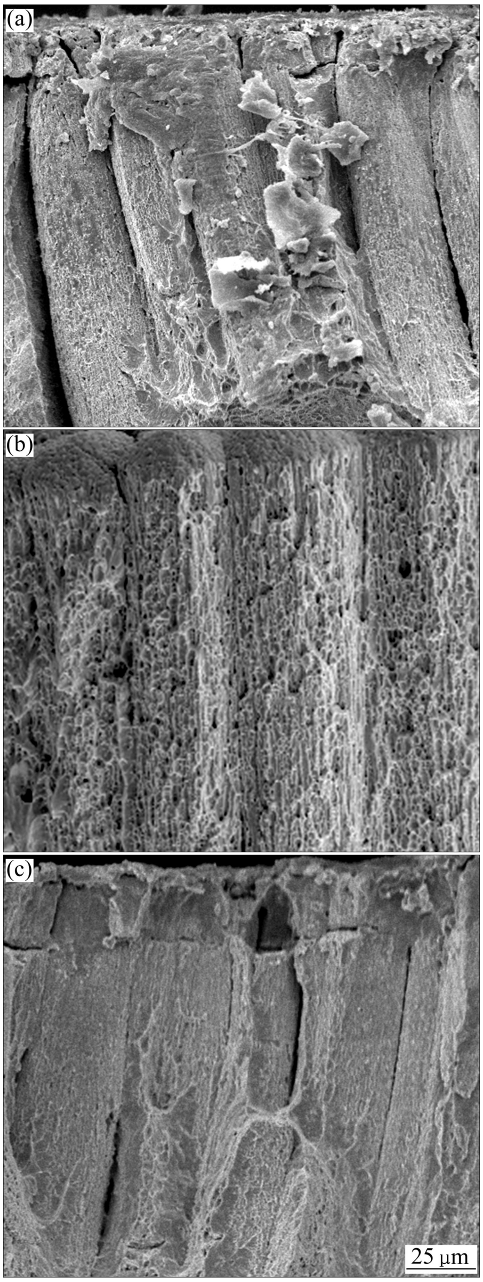

Fig. 3 SEM images of fractured surface of NiCoCrAl alloy sheet heat treated at different temperature for 40 min: (a) 950 ºC; (b) 1050 ºC; (c) 1150 ºC

Fig. 3(a), compared with the fractured surface of the untreated specimen shown in Fig. 2(a). As the heat treatment temperature increased, the width of the gap parallel to the columnar structure obviously reduced, especially no gap was observed on the fractured surface for the specimen heat treated at 1050 °C because of the element diffusion. The heat treatment at 1050 °C was favorable to improve the interface bonding between columnar structures due to the disappearance of the gap, which resulted in that the fracture mode was changed from inter-columnar fracture to trans-granular fracture. As shown in Fig. 3(c), the fractured surface for the specimen heat treated at 1150 °C was relatively flat, only with little dimple fracture feature, which was unfavorable to the ductility of the alloy sheet. The poor ductility was probably attributed to the grain over-growth up during 1150 °C heat treatment.

In order to investigate the effect of the heat treatment on microstructure, the phase compositions of the NiCoCrAl alloy sheet before and after the heat treatment were studied. The XRD patterns of the specimens heat treated at 950 and 1150 °C did not show the obvious change compared with the XRD pattern of the untreated specimen and the specimen heat treated at 1150 °C showed obvious distortion due to the high temperatures. The phase compositions of the NiCoCrAl alloy sheet before and after the heat treatment at 1050 °C were investigated and shown in Fig. 4. The phase analysis revealed that the γ-Ni phase in the NiCoCrAl alloy sheet before the heat treatment was crystal and the balance in the NiCoCrAl alloy sheet was amorphous. In comparison with the NiCoCrAl alloy sheet before the heat treatment, the Ni_3Al phase occurred in the NiCoCrAl alloy sheet after the heat treatment. The formation of the new phase (Ni_3Al phase) is favorable to the interface bonding because of the element diffusion, which led to that the fracture mode was changed from inter-columnar fracture to trans-granular fracture [15].

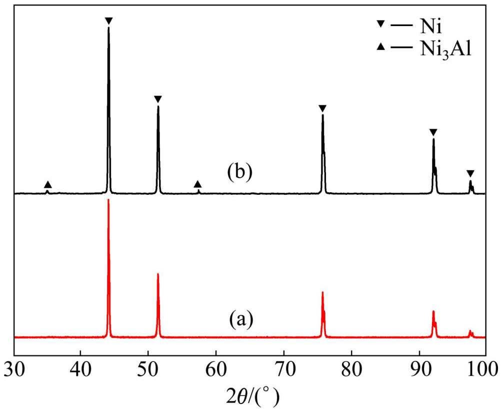

Fig. 4 XRD patterns of NiCoCrAl alloy sheet before (a) and after (b) heat treatment at 1050 °C

The SEM images of the fractured surface of the NiCoCrAl alloy sheet after the heat treatment at 1050 °C for different time are shown in Fig. 5. The gap width of the fractured surface of the specimen heat treated at 1050 °C for 30 min was reduced, as shown in Fig. 5(b), compared with the fractured surface for the specimen before the heat treatment shown in Fig. 5(a). The NiCoCrAl alloy sheet was fractured still along the interface between columnar structures. As the heat treatment time increased up to 60 min, no gap was observed on the fractured surface and the obvious transgranular fracture of the columnar structure was detected readily, as shown in Fig. 5(c), which indicated that the interface bonding strength between columnar structures was equivalent to the strength of the columnar

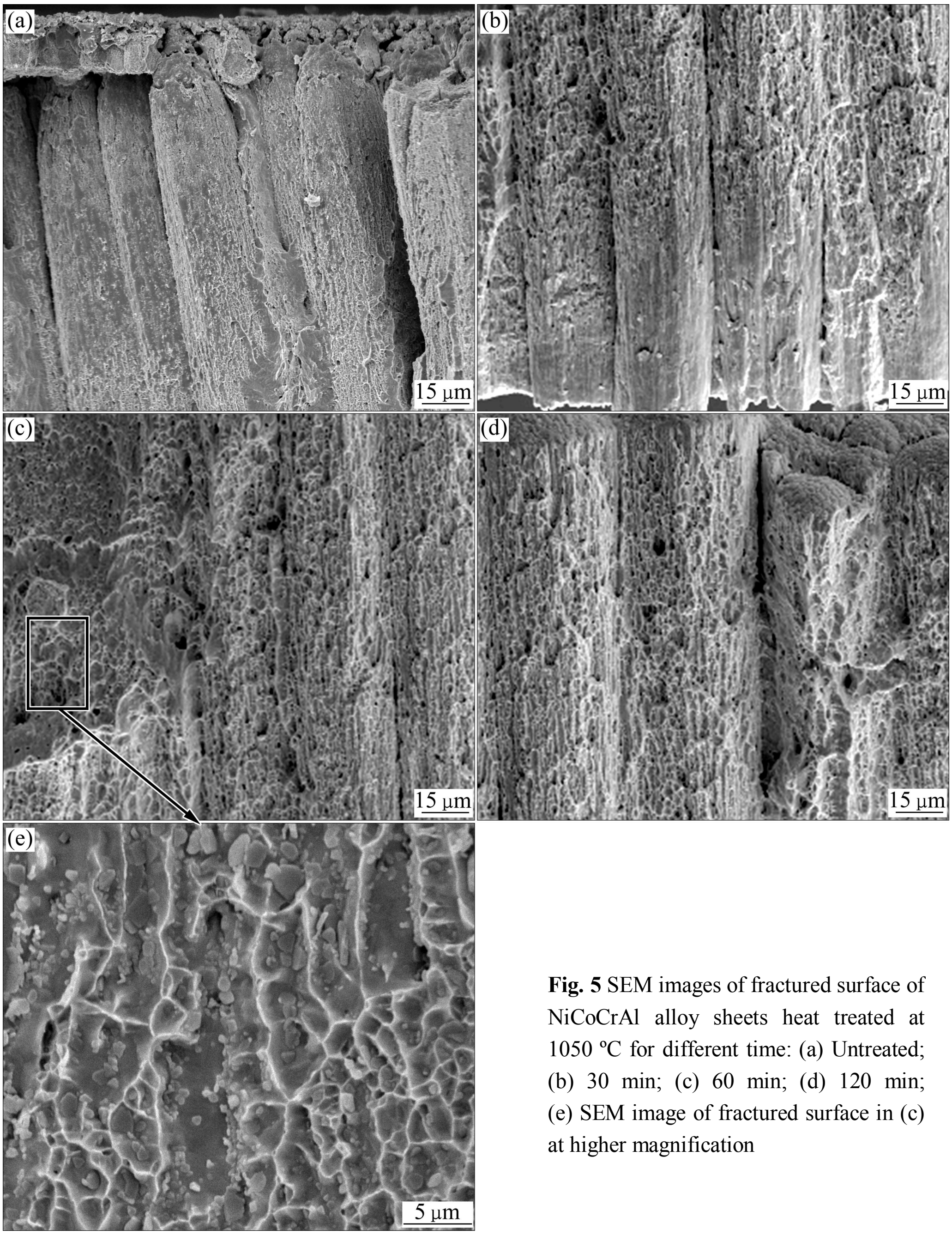

Fig. 5 SEM images of fractured surface of NiCoCrAl alloy sheets heat treated at 1050 °C for different time: (a) Untreated; (b) 30 min; (c) 60 min; (d) 120 min; (e) SEM image of fractured surface in (c) at higher magnification

structure. The fractured surface did not show an obvious change with increasing heat treatment time to 120 min as shown in Fig. 5(d), compared with the fractured surface treated for 60 min, whereas the obvious deformation of the macrostructure occurred. Furthermore, the significant plastic deformation in the transgranular fracture region of the columnar structure was detected readily, as shown in Fig. 5(e), which revealed good boundary bonding strength between the particles [16].

3.2 Tensile strength

Figure 6 shows the typical stress—strain curves of the NiCoCrAl alloy sheet before and after the heat treatment at 1050 °C for different time. It could be seen from the typical stress—strain curve of the NiCoCrAl alloy sheet before the heat treatment that the untreated specimen showed an absolute brittle fracture without plastic deformation. The tensile strength and failure strain were (644±45) MPa and 0.38%, respectively. For the specimen heat treated for 30 min, the slight plastic deformation was observed, and the tensile strength and failure strain were improved to be (662±37) MPa and 1.46%, respectively. As the heat treatment time further increased up to 60 min, the failure strain increased significantly to 6.2% and the tensile strength obviously increased to (705±51) MPa, whereas the tensile strength and failure strain were reduced to (671±47) MPa and 4.17%, respectively, compared with the specimen treated for 60 min as the heat treatment time further increased to 90 min. The increase in the tensile strength and elongation was mainly attributed to the improvement in the interface bonding between the columnar structures [17].

It was recognized that the presence of the residual stress was generally harmful to the interface bonding [17,18]. In this work, X-ray diffractometry was used to research the effect of the heat treatment on the residual stress. The diffraction data of 331 crystal face of the γ-Ni phase were used based on the $\sin^2\psi$ method to investigate the effect of the heat treatment on the residual stress [19]. The spacing of specific sets of crystallographic planes was measured, and elastic strain (ε) was calculated by comparing the measured planar spacing (d) with the strain-free lattice spacing (d_0).

$$\varepsilon=\frac{d-d_0}{d_0}=\frac{\Delta d}{d_0}=-\frac{\Delta 2\theta}{2\tan\theta_0} \tag{1}$$

The residual stress could be expressed by:

$$\sigma=\frac{-E}{2(1+v)}\frac{1}{\tan\theta_0}\frac{\pi}{180}\frac{\partial(2\theta)}{\partial(\sin^2\psi)}=K\frac{\partial(2\theta)}{\partial(\sin^2\psi)} \tag{2}$$

where θ_0 is the diffraction angle of crystal with free stress; θ is the diffraction angle of the crystal face located at the angle ψ of the crystal face with incident ray; K is the stress constant; ψ is the angle of the normal line of crystal face with diffraction face. Thus, one can use diffraction data from the specific planes to calculate the residual stress [19] based on the change curve of lattice spacing with $\sin^2\psi$, as shown in Fig. 7. The residual stress of the NiCoCrAl alloy sheet before the heat treatment at 1050 °C for 60 min was calculated to be (−34.2±3.5) MPa. After the heat treatment at 1050 °C for 60 min, the residual stress was reduced significantly to (−3.7±1.2) MPa. The residual stress was mostly caused by the uneven microstructure of the alloy sheet, especially uneven distribution of the intergranular gaps. When the NiCoCrAl alloy sheet was removed from the substrate, there was a decreasing tendency of the inter-columnar gap sizes due to shrinkage. The zones with

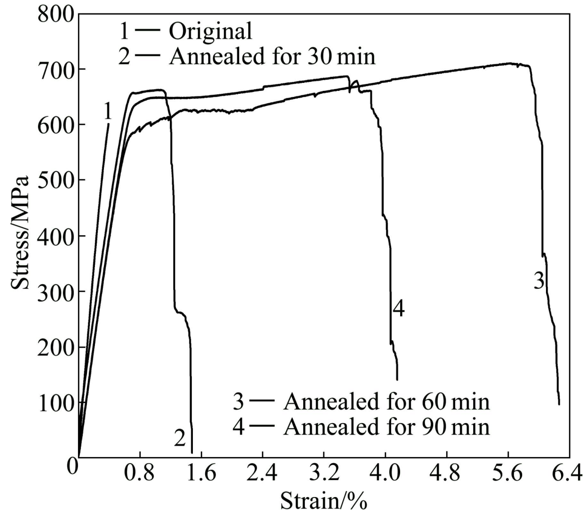

Fig. 6 Typical stress—strain curves of NiCoCrAl alloy sheet before and after heat treatment at 1050 °C for different time

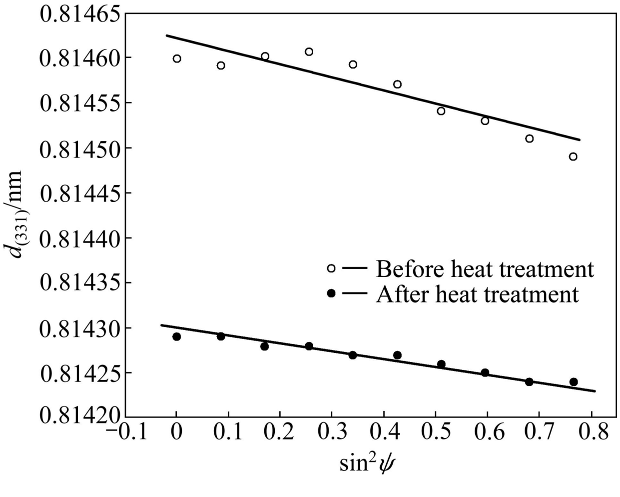

Fig. 7 Change curve of distance between crystal faces with $\sin^2\psi$

different inter-columnar porosities had different shrinkage degrees in the NiCoCrAl alloy sheet, which caused the residual stress. During the 1050 °C heat treatment, the microstructure of the alloy sheet tended to be uniform due to element diffusion, which resulted in the improving interface bonding and the decreasing intergranular gaps. The improvement in the interface bonding was confirmed by the reduction in the residual stress. Therefore, the reduction in the residual stress was also favorable to improve the tensile strength of the NiCoCrAl alloy sheet.

4 Conclusions

1) The width of the gap parallel to the columnar structure for the NiCoCrAl alloy specimen heat treated at 950 °C for 40 min is obviously increased compared with the fractured surface of the untreated specimen. As the heat treatment temperature increases, the width of the gap obviously reduces, especially no gap is observed for the specimen heat treated at 1050 °C.

2) The NiCoCrAl alloy specimen heat treated at 1150 °C shows the poor ductility due to the grain overgrowth. However, the specimen heat treated at 1050 °C shows the improved mechanical properties.

3) The formation of the Ni_3Al phase is favorable to improve the interface bonding between NiCoCrAl columnar grains. The increase in the tensile strength and elongation is attributed to the improvement in the interface bonding between the columnar structures.

4) The residual stress of the NiCoCrAl alloy sheet after the heat treatment at 1050 °C for 60 min is reduced significantly from (−34.2±3.5) MPa to (−3.7±1.2) MPa, which also confirms that the interface bonding is improved by the heat treatment.

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热处理对 EB-PVD 制备的 NiCoCrAl 薄板的微观结构和拉伸强度的影响

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摘　要：采用电子束物理气相沉积技术制备 NiCoCrAl 合金薄板，研究热处理对合金薄板的微观结构和拉伸强度的影响。制备态的合金薄板具有柱状晶结构，柱状晶间存在间隙。经 1050 °C 热处理，柱状晶间的间隙消失，从而导致柱状结构间的结合强度提高；新相 Ni_3Al 在合金中析出，这有助于提高柱状结构间的结合强度。热处理后，合金薄板中残余应力减少，证明薄板中柱状结构间的界面结合有改善。柱状结构间结合强度的提高最终导致合金薄板的拉伸强度和伸长率的提高。

关键词：镍基合金；热处理；微观结构；力学性能；物理气相沉积

(Edited by LI Xiang-qun)