XRD investigation of thermostability of TiB₂/Al deformation layer introduced by shot peening

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Abstract

The thermostability of 6351Al and TiB₂/6351Al during heat treatment was investigated utilizing X-ray diffraction. Domain size and microstrain were calculated by modified Warren–Averbach method and Voigt method, respectively. Results showed that the textures of both specimens were randomized by shot peening and did not appear again after all heat treatments. Domains had grown during heat treatments, and the growth of composite was slower than that of alloy. The microstrain of composite is easier to release than alloy. Higher density dislocation around reinforcements promotes recrystallization while further domain growth was retarded. The similar variation of microstrain and FWHM shows that the dominant factor to affect FWHM is microstrain. The thermostability of composite is higher than that of alloy.
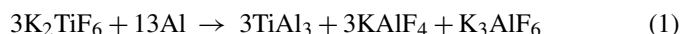© 2007 Elsevier B.V. All rights reserved.

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1. Introduction

Metal matrix composites have been widely concerned because of excellent properties such as high specific strength and stiffness, etc. [1]. In the process of shot peening (SP), a great amount of balls at high velocity impact on the surface of sample which causes elastic and plastic deformation. As a result, domain refinement, and microstrain, etc., are introduced to the deformation layer. The fatigue strength [2] and stress corrosion resistance [3], etc., of specimen are then improved after SP. The thermostability of composites at elevated temperature is very important to retain these properties. X-ray diffraction (XRD) is an important method to characterize microstructure because of its reliability and non-destruction to sample. Using line profile analysis, microstructure can easily be obtained. However, little work has been conducted on the thermostability of composites after SP together with heat treatment (HT) so far. It is significant to investigate microstructure and thermostability of the deformation layer by XRD through which to find a way to improve the properties of composites.

2. Experimental

Ten volume percentage of TiB₂/6351Al in situ composite and 6351Al alloy were investigated after SP and HT. During the synthesis of the in situ composite, mixed salts of potassium hexafluorotitanate (K_2TiF_6) and potassium tetrafluororate (KBF_4) are introduced into a stirred aluminum melt with an atomic ratio in accordance with Ti/2B. Exothermal reactions between the salts and the molten aluminum take place according the following sequences [4,5]:

$$3K_2TiF_6 + 13Al \rightarrow 3TiAl_3 + 3KAlF_4 + K_3AlF_6 \qquad (1)$$

$$2KBF_4 + 3Al \rightarrow AlB_2 + 2KAlF_4 \qquad (2)$$

$$AlB_2 + TiAl_3 \rightarrow TiB_2 + 4Al \qquad (3)$$

Before SP, both specimens were heat treated at the same conditions: solution treatment at 530 °C for 110 min, then quenched in water at room temperature, and finally aging at 170 °C for 6 h. Young's modulus of the composite and alloy are 80 GPa and 69 GPa, and yield strength 370 MPa and 275 MPa, respectively. The SP parameters include: air pressure 5.88×10^5 Pa, SP time 5 min, the SP media is glass balls with 0.5 mm average diameter. The arc height of Almen specimen under such SP conditions is 0.22 mm. In order to investigate the thermostability of

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alloy and composite after SP, both materials were heat treated at 100–400 °C of interval 25 °C using a single specimen each, holding time is 5 min at each stage. Postmortem XRD experiments were conducted on the same zones after each stage cooling the specimens to the room temperature.

The diffraction profiles of both specimens were measured by Dmax/rc diffractometer with Cu Kα radiation, voltage 40 kV and current 100 mA. The microstrain and domain size of the deformation layer were calculated by Voigt method [6] and modified Warren–Averbach method [7,8], respectively.

For Voigt method, the relationship of integral breadths is given by:

$$\beta_G^{h2} = \beta_G^{f2} + \beta_G^{g2}, \qquad \beta_C^{h} = \beta_C^{f} + \beta_C^{g} \tag{4}$$

where subscript G and C denote the Gaussian and Cauchy components, and superscript h, f, g denote the measured line profile, the structurally broadened profile and the instrumental profile, respectively. It is assumed that the Gaussian contribution arises from microstrain. The microstrain is given by:

$$\varepsilon = \frac{\beta_G^f}{4\tan(\theta)} \tag{5}$$

In this paper, Al(1 1 1) and Si(1 1 1) profiles were measured as h and g profiles, respectively. For both profiles, the backgrounds were subtracted and $k_{\alpha2}$ profiles were striped according to Rachinger method.

According to T. Ungar [7,8], the equation of modified Warren–Averbach is:

$$\ln A(L) \cong \ln A^s(L) - \rho B L^2 \ln\left(\frac{R_e}{L}\right)(K^2\bar{C}) + O(K^4\bar{C}^2) \tag{6}$$

where $A(L)$ is the real part of the Fourier coefficients, $A^s(L)$ the size Fourier coefficient, $B = \pi b^2/2$, R_e the effective outer cut-off radius of dislocations and O stands for higher order terms in $K^4\bar{C}^2$. L is the Fourier length defined as $L = na_3$, where $a_3 = \lambda/2(\sin\theta_2 - \sin\theta_1)$, n are integers starting from zero and $(\theta_2 - \theta_1)$ is the angular range of the measured diffraction profile. $K = 2\sin\theta/\lambda$ and $K = g$ at the exact Bragg position. The average contrast factors $\bar{C}$ can be calculated from reference [8,9]. The size parameter corresponding to the Fourier coefficients is denoted by L_0. It is deduced from the size Fourier coefficients $A^s(L)$ versus L by least-squares method, according to the formula provided by W. Yuming et al. [10]:

$$A^s(L) = a - \frac{L}{L_0} \tag{7}$$

where a is the quantity expressing the "hook" effect. And it gives the area-weighted mean column length.

In order to get $A(L)$, stokes deconvolution [11] was conducted, taking the measured profile of the standard annealed sample as the instrument broadening profile. The standard sample has the same composition of 6351Al and annealed thoroughly at 450 °C for 6 h.
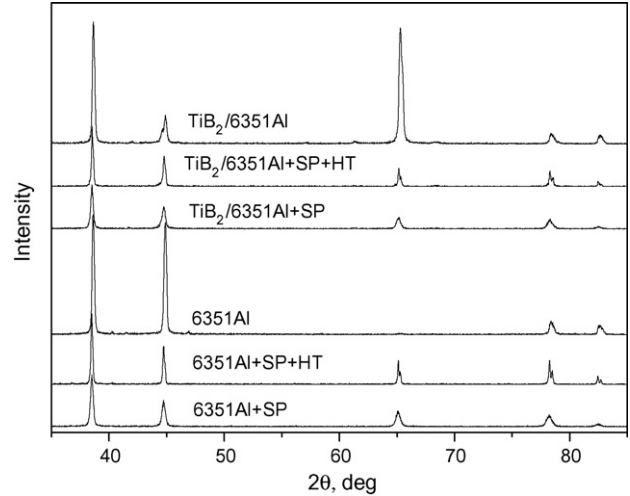

Fig. 1. Change of XRD patterns (SP: air pressure 5.88×10^5 Pa, 5 min; HT: heat treatment at 100–400 °C, interval 25 °C, 5 min holding time for each stage).

3. Results and analysis

Fig. 1 shows the XRD patterns of alloy and composite after different treatments (unpeened, SP and SP + HT). It can be seen that the unpeened samples of alloy and composite have severe textures. While after SP, all textures were randomized, and did not appear after HT. Using Harris method [12], the texture coefficient (TC) was calculated:

$$TC_{(hkl)} = \frac{I_{(hkl)}/I_{0(hkl)}}{(1/n)\sum I_{(hkl)}/I_{0(hkl)}} \tag{8}$$

where, $I_{(hkl)}$ and $I_{0(hkl)}$ are integral intensity of (hkl) peak of samples and aluminum powder, respectively. And n is the number of diffraction peaks. The texture coefficient of (2 2 0) peak is 2.77 for composite. For alloy, the texture coefficient of (1 1 1) and (2 0 0) peak is 1.42 and 2.46, respectively.

In the process of SP, as balls with high energy impacting on the surface of sample from different directions, elastic and plastic deformation is introduced. As the result, grains are refined and hence become equiaxial [13,14]. Moreover, the grain boundaries increased. The refined grains and increased boundaries make grains easy to rotate in further deformation, through which the textures are randomized in the uppermost surface of samples. The change of grain size and boundaries caused by SP is illustrated in Fig. 2. The grown grains still remain equiaxial during HT. Therefore the XRD pattern of both specimens showed no texture after HT.

Domains had grown as the temperature increased, as shown in Fig. 3. Using modified Warren–Averbach method, domain size of alloy and composite were calculated. After SP, they were 37 nm and 58 nm, while grew to 580 nm and 240 nm after HT, respectively. In Fig. 3, the domain size of each HT stage was normalized by the original values (37 nm for alloy and 58 nm for composite). It can be seen that the growth of alloy is faster than that of composite during HT. For alloy and composite, the domains had grown to 16 ± 1 and 4 ± 1 times after HT, respectively.