XRD Investigation of Thermal Relaxation Behavior of Microstructure in TiB_2/Al Deformation Layer Introduced by Shot Peening

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Abstract: The investigation of microstructure thermostability and residual stresses relaxation of TiB_2/Al composite were carried out via X-ray diffraction and XRD line profile analysis. Results showed that with increasing the heating temperature and time, domain size increased and microstrian as well as dislocation density decreased, respectively. During annealing, the peened microstructures recovered and recrystallized. Because of the pinning effect of the reinforcement particles, the growth of domain size of alloy was faster than that of composite during annealing. For alloy and composite, the domains had grown to 16 and 4 times after annealing, respectively. Using modified Warren-Averbach method, the calculated domains of alloy and composite grow to 16 and 4 times after HT. Using Voigt method, the calculated domains of alloy and composite grow to 12 and 8 times after HT.
Keywords: thermal relaxation, Line profile analysis, shot peening, TiB_2/Al composite.

Introduction

Metal matrix composites have wide application prospects because of their good combination of excellent properties. TiB2/6351Al composite combines the metallic properties with the ceramic's reinforcement characteristics, leading to greater strength, modulus and hardness as well as higher service temperature capabilities [1-3]. In the process of shot peening (SP), a great number of balls impact on the surface of sample with high velocity, which causes elastic and plastic deformation. In terms of microstructure, the change of domain size, microstrain and dislocation density are also introduced to the deformation layer. It is well known that the fatigue strength and stress corrosion resistance of specimen are improved after shot peening. However, the physical metallurgical processes such as recrystallization and grain growth which happen at high temperature can deteriorate the properties of shot peened workpieces. Therefore, the thermostability of composite at elevated temperature is very important for retaining high properties of composites after shot peening.

X-ray diffraction line profile (XRDLPA) is a powerful method to describe the microstructures of crystalline materials because of its reliability and non-destruction to sample. Several new methods of XRDLPA have recently been developed through which reliable results of microstructures can be obtained easily [4-6]. However, little paper has been conducted on the thermostability of composites after shot peening using X-ray diffraction line profile (XRDLPA). This work investigates the microstructure and thermostability of in situ TiB_2/6351Al and 6351Al alloy deformation layers after SP by XRDLPA.

Experiments

The shot peened layers of the TiB_2/6351Al composite (in situ, 10 vol.% TiB_2) and the 6351Al alloy were investigated during heat treatment (HT). The composite was synthesized according to Refs. [2,7]. Before SP, both specimens were heat-treated at the same conditions: solution treatment at 530°C for 110 min, then quenched in water, and finally aging at 170 °C for 6 h. SP treatments were carried out according to the conditions: air pressure 5.88×10^5 Pa, SP time 5 min, ceramic beads with average diameter 0.5 mm. The dimensions of all specimens were 20mm×20mm×5mm and the SP treated surface was 20×20mm. The shot peening intensity was 0.22 mmA under such SP conditions. In order to investigate the thermostability of recrystallization behaviors of 6351Al alloy and TiB2/6351Al composite after SP, continuous heating annealing were carried out on 6351Al alloy and the TiB_2/6351Al composite samples. The regime of continuous heating annealing was heating samples during 100-400 °C of interval 25°C for one step and 5 min for each step holding time. Postmortem XRD investigations were conducted on the same zones after cooling from high temperature to room temperature during annealing.

The diffraction profiles of both specimens were measured by Dmax/rc diffractometer with Cu-*Kα* radiation, voltage 40 kV and current 100 mA. The domain size and microstrain of the deformation layer were calculated by Voigt method [8] and modified Warren-Averbach method [4,9], respectively.

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

$$\beta_G^{h^2}=\beta_G^{f^2}+\beta_G^{g^2}\ ,\quad \beta_C^h=\beta_C^f+\beta_C^g \tag{1}$$

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 Cauchy component of the *f* profile is solely due to domain size and that the Gaussian contribution arises from microstrain. In Voigt method, the microstrain and domain size are given by:

$$\varepsilon=\beta_G^f\big/4\tan(\theta)\quad ,\quad D=\lambda\big/\beta_C^f\cos(\theta) \tag{2}$$

In this paper, Al(111) and Si(111) profiles were measured as *h* and *g* profiles, respectively. For both profiles, the backgrounds were subtracted and $k_{\alpha 2}$ profiles were striped according to Rachinger method.

According to T. Ungár [4,9], the basic equation of modified Warren-Averbach (MWA) method is

$$\ln A(L)\cong\ln A^s(L)-\rho BL^2\ln(\mathrm{R_e}/L)(K^2\overline{C})+O(K^4\overline{C}^2) \tag{3}$$

where *A(L)* is the real part of the Fourier coefficients, $A^s(L)$ is the size Fourier coefficient, $B=\pi b^2/2$, R_e is 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 [5,9]. 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 Wang [10]:

$$A^s(L) = a - L / L_0 \tag{4}$$

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 550°C for 6 h. For all profiles, the backgrounds were subtracted.

Result and discussion

It is well known that the domain size grows with increasing the heating temperature and the heating time. Normalized domain size of 6351Al alloy and TiB2/6351Al composite were calculated using modified Warren-Averbach method and Voigt method, as shown in Figure 1. The domain size of each HT stage was normalized by the original values (37nm for 6351Al alloy and 58nm for TiB_2/6351Al composite). It can be seen that no matter using Warren-Averbach method and Voigt method, the increments of 6351Al alloy domain size and TiB_2/6351Al composite domain size are not obvious in the initial stage of heating due to the low heating temperature and the short heating time in this stage. In terms of the whole heating process, the growth rate of 6351Al alloy domain is much larger than that of TiB_2/6351Al composite. Using modified Warren-Averbach method, the calculated domains of alloy and composite grow to 16 and 4 times after HT. However, Using Voigt method, the calculated domains of alloy and composite grow to 12 and 8 times after HT. The difference between this two calculated results is due to applying different methods to calculate the domain size. The area-weighted mean column length is obtained via Modified Warren-Averbach method and mean column length in diffraction direction is obtained via Voigt method.

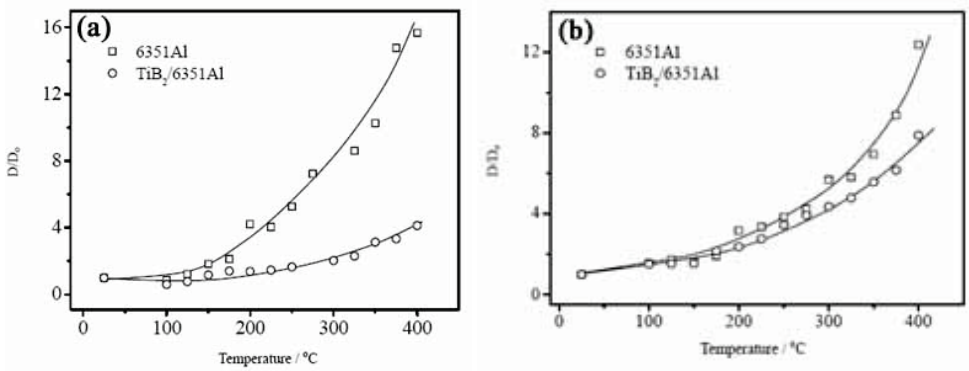

Fig. 1. Variations of domain size during continuous heating, calculated using (a) MWA method and (b) Voigt method.

(D: domain size at *T* temperature, D_0: original domain size before heating)

As the strength of reinforcement is much higher than the strength of matrix, the reinforcement can increase the deformation of matrix around reinforcement itself and increase store energy during deformation process. When the reinforcement size is large, the reinforcement particles in matrix composites provide more nucleation sites for new

recrystallized grains [12-14] and the nucleation is promoted by the addition of reinforcements [15]. However, when the reinforcement size is small, even though there are a lot of store energies existing around reinforcement in matrix composites, further growth of domains are retarded [15] because of the pinning effect of the refine reinforcement particles, which hinder the recrystallization process. The reinforcement particles in this TiB_2/6351Al composite are small, so at the same HT conditions, the domain size of TiB_2/6351Al composite is relative stable than that of 6351Al alloy.

Another change caused by continuous heating annealing is the decrease of the microstrain. Figure 2a shows the microstrain of both samples calculated from Al(311) reflections using Voigt method. There are three stages of both materials during continuous heating annealing: (1) slight decrease stage, about 25°C-400°C, (2) sharp decrease stage, about 225°C-300°C and (3) stable stage, about 300°C-400°C. In the first two stages, the decrease of composite is always rapid than that of alloy. While in the third stage, the value of microstrain of composite is little larger than that of alloy. This is mainly due to the thermal mismatch between the matrix and the reinforcements when cooling from high temperature to room temperature. Furthermore, the temperature range of TiB_2/6351Al composite sharp decrease stage is lower than the temperature range of 6351Al alloy sharp decrease stage, which means the microstrain of composite more easily releases than alloy. In shot peened alloy and composite, dislocations are not only the main defects of material but also they are the major reason to the microstrain. The decrease of dislocation density causes the decrease of microstrain. However, the increments of domain size need time to complete via migration of grain and subgrain boundaries. Therefore, even though the microstrain decreases sharply between 225 °C and 300 °C, the domain size increases slowly during this period.

Figure 2b shows the Al (311) FWHM changes of the 6351Al alloy and TiB_2/6351Al. It is interesting that the variation of FWHM is similar to that of microstrain for both specimens. In this paper, the FWHM of instrumental was striped from the measured line profile, thus the obtaind FWHM comes from the specimens only.

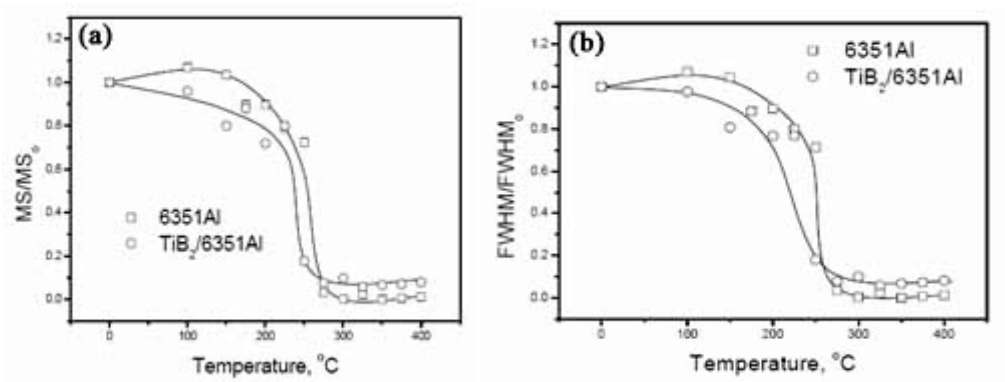

Fig. 2(a) Variations of microstrain during continuous heating via Voigt method (MS: Microstrain at *T* temperature, MS_0: original Microstrain before heating)
2(b) Al(311) full width at half maximum variations during continuous heating annealing (FWHM: Full width at half maximum at *T* temperature, $FWHM_0$: Full width at half maximum before heating)

In deformed metals, microstrian is mainly comes from dislocations because not only they

are always the main defects but also they are the major components or can play an important role in other defects [16,6]. Therefore, the change of microstrain depends mainly on change of dislocation density. The dislocation density can be increased extremely by SP. The reinforcement particles in composite always act as sink sources [17] of dislocations during repeated deformation. It has been reported that the high-density dislocations around the reinforcement particles promote recrystallizaiton[14,18]. The growth of recrystallization nucleus largely depends on the stored energy [19]. High dislocation density means high store energy. During heating annealing, the stored energy is released for recrystallizaiton and further domain growth. The higher density dislocations and more nucleus around reinforcement particles make the microstrian easier to release.

The recrystallization of deformed metal is a thermodynamic process and the heating temperature and the heating time are the two main factors affecting this process. The sharp decease of microstrain during 225°C-300°C represents the recrystallization take place in this period and the corresponding time range is 30-45 min. The phenomenon of the increment of domain size is less obvious than the decrease of microstrain. Because of the pinning effect of the reinforcement particles, the growth of alloy domain size is faster than that of composite domain size during annealing, which means the thermostability of composite at elevated temperatures is higher than that of alloy.

Conclusions

Using XRDLPA, the recrystallization behaviors of shot peened 6351Al alloy and TiB_2/6351Al composite surface layers have been investigated in the condition of continuous heating annealing and isothermal annealing. In continuous heating annealing process, the domain size increased and the microstrain as well as dislocation density decreased with increasing heating time and heating temperature. Because the movements of grain or subgrain boundaries were impeded by the reinforcement, the growth rate of domain size of alloy was faster than that of composite during annealing. For alloy and composite, the calculated domains have grown to 16 and 4 times via Modified Warren-Averbach (MWA) method and 12 and 8 times via Voigt method. The high-density dislocations around the reinforcement particles promoted recrystallizaiton at early stage of continuous heating annealing, so the microstrain of composite more easily released than alloy during that period.

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