

Fe–Al Phase Formation Studied by Internal Friction during Heating Process *

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We systematically investigate the internal friction properties of a Fe–(43 at.%)Al powder mixture compact during the heating process with the expectation to understand the phase formation and transition process. Three internal friction peaks are successively observed during the heating process from room temperature to 750°C, but almost completely disappear in the subsequent cooling process. Three internal friction peaks exhibit obvious measuring frequency dependence, which increases with decreasing the frequency. The first internal friction peak originates from the micro-sliding of weak bonding interface between Al particles corresponding to a recrystallization process of deformed Al particles. The second internal friction peak is attributed to a phase formation process associated with the formation of the intermediate phase Fe₂Al₅. The third internal friction peak is considered to result from the formation of the FeAl intermetallic compound owing to the reaction of Fe₂Al₅ and residual Fe initiated by a dramatic thermal explosion reaction.

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Intermetallic compounds have increasingly attracted attention because of their excellent physical and mechanical properties of critical importance in a variety of applications.^[1] Monolithic intermetallic materials such as Ni₃Al, NiAl, Fe₃Al, FeAl, Ti₃Al and TiAl are typical representatives. Among the intermetallics, iron aluminides are especially interesting due to their low density, low material cost and good high temperature mechanical properties. In addition, attributed to the formation of dense and adherent protective Al₂O₃ scale on the surface of the materials, iron aluminides have good high temperature corrosion resistance in oxidizing and sulfidizing environments.^[2–4] In recent years, a variety of methods have been developed to manufacture iron aluminides as the examples of melting, roll compaction and mechanical alloying. The powder metallurgy technique, including powder compaction and sintering, may sometimes be the only reasonable candidate to meet specific applications, which offer additional advantages such as net-shape processing, better control of microstructure, and cost-effectiveness.^[2]

Heating process of the Fe–Al system accompanied with the phase formation and transition is critical to determine the final product quality that a number of studies have been carried out to understand the microstructure evolution during heating process by various technical methods. Gedeonishvili investigated the phase transition process of elemental powders of Fe and Al at various heating rates by dilatometric experiments.^[2] Charlot studied the mechanically activated synthesis in the Fe–Al system by x-ray diffraction.^[5] Pocheć investigated the phenomena preceding and accompanying the SHS reaction between

Fe and Al elemental powders using SEM and XRD analyses.^[6] Sina studied the formation of Fe–Al system during the heating of Fe–(40 at.%)Al powder mixture using a differential scanning calorimeter.^[7] Naoi experimentally observed the solid-state reactive diffusion between Fe and Al using Al/Fe/Al diffusion couples prepared by a diffusion bonding technique.^[8] Even though many investigations as mentioned above have been widely performed, it is difficult to track phase transformation process by classical methods, such as SEM and XRD owing to the high speed of the high temperature synthesis and strongly exothermic nature of Fe–Al elemental powder mixture during heating process. Moreover, a general acknowledged conclusion has not been drawn as yet due to the complexity of phase transformation of Fe–Al system during heating process.

In the present work, the internal friction technique associated with the thermal analysis is applied to further investigate the phase transformation of the Fe–Al powder compact during the heating process. It is well acknowledged that internal friction is so sensitive to response the microstructure change that can provide information at an atom scale, such as phase transition, solid defects evolution, grain boundary relaxation, and dislocations evolution.^[9]

The two elemental sphere-shaped powder particles were commercially pure iron (6–10 μm, 99.5% purity) and pure aluminum (25 μm, 99.9% purity) supplied from Alfa and Macklin, respectively. The mixture of Fe and Al powder with the chemical composition Fe–(43 at.%)Al in the range of B2 crystal structure was obtained in a single-tank planetary high-energy ball mill (Germany, Fritsch-Pulverisette 6). The mechani-

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cal mixing of the two powder particles without grinding ball was carried out at the rotation speed 200 rpm for 2 h in a vacuum condition of 10^{-3} Pa. The mixture was pressed to form rectangular bars with the dimensions of $65 \times 5 \times 1.2$ mm³ through a uniaxial compaction under 400 MPa in a rectangular steel die without any assistance of binder and lubricant to avoid impurities induced.

The internal friction (IF, Q^{-1}) and relative dynamic modulus (RDM) of the powder compact of Fe–Al mixture were measured using a multi-function internal friction apparatus (MFIF-I). The apparatus mainly consisted of an inverted torsion pendulum, a temperature programmer, a photoelectron transformer and an automatic computer controlled system to operate the whole measurement and to process the data in real time. The details of the apparatus can be found in Ref. [10]. The measurement was carried out from room temperature to 750°C in a vacuum atmosphere of 10^{-3} Pa. The strain amplitude of 20×10^{-6} is adopted, and measurement frequencies of 0.5, 1.0, 2.0 and 4.0 Hz are employed. For comparative purposes, the internal friction measuring of the pure Al powder compact is also conducted with the same measuring parameters as the Fe–Al system. With the aim at determining the nature of the reactions and the temperature of various reactions of the Fe–Al powder compact, the thermal analysis was performed using a thermogravimetry differential scanning calorimeter (TG-DSC: STA449F3, Netzsch, Germany) under a nitrogen atmosphere with a heating rate of 5°C/min.

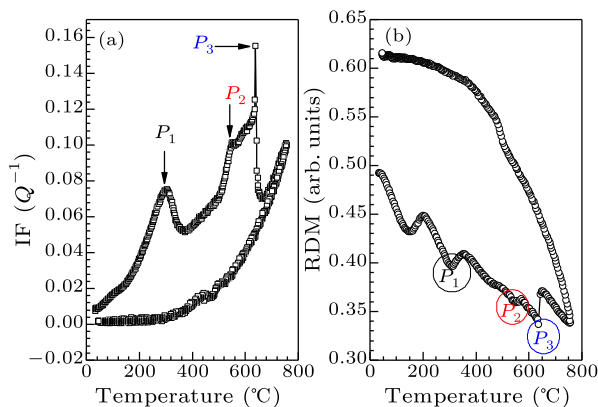


Fig. 1. Dependence of IF (a) and RDM (b) on temperature during heating and subsequent cooling process of Fe–Al powder compact.

Figures 1(a) and 1(b) show the typical IF and RDM characteristics of the Fe–Al powder compact during the heating process from room temperature to 750°C for the first cycle. The heating rate and measuring frequency adopted are 1°C/min and 0.5 Hz. The most outstanding features are the appearance of three IF peaks as marked by the arrows with different colors existing at around 305°C (designated P_1), 560°C designated P_2) and 645°C (designated P_3) corresponding to the heating process. However, the three IF peaks

completely disappear during the subsequent cooling process. In accordance with the appearance of the three IF peaks, the RDMs exhibit a local minimum as signed by the circles with various colors, indicating the micro-structure transition of the Fe–Al powder compact in the heating process. To further understand the mechanism of the peaks, the measuring frequency dependence of IF is described in Fig. 2. It can be observed that three peaks increase with the decreasing measuring frequency. These reverse dependences between IF peak and measuring frequency are in accordance with those of IF peak originated with the phase transition.^[9]

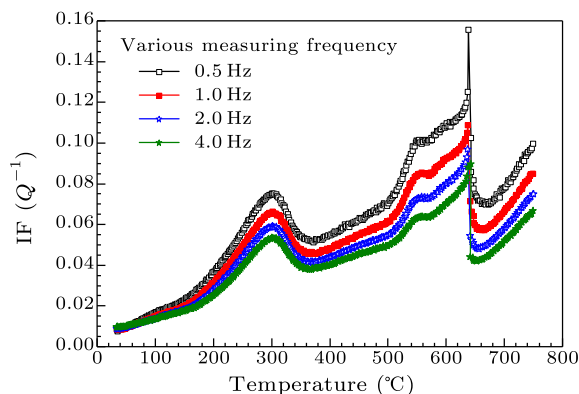


Fig. 2. Dependence of IF on measuring frequency of Fe–Al powder compact.

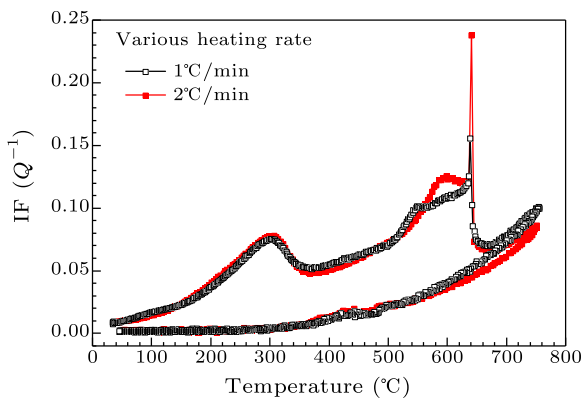


Fig. 3. Dependence of IF on heating rate of Fe–Al powder compact.

The dependence of heating rate on the IF characteristics of the Fe–Al powder compact during the heating process is characterized in Fig. 3. It can be found that the slight influence on the P_1 and P_3 IF peaks can be neglected, especially the P_3 peak. However, the peak location of the P_2 peak is strongly controlled by the heating rate, which shifts towards higher temperature with increasing the heating rate. The P_2 peak temperature location is around 595°C at the heating rate 2°C/min, much higher than 560°C at the heating rate of 1°C/min.

A comparison of the IF properties between Fe–Al and pure Al powder compact is depicted in Fig. 4. The first cycle measurement was carried out from room

temperature to 450°C at the frequency 1.0 Hz. It can be noticed that the P_1 peak existing in the Fe–Al powder compact are almost same as that appearing in pure Al powder compact except for the difference of peak height. The peak temperature locations in Fe–Al and pure Al are well in accordance, both existing at around 305°C. For pure Fe powder compact, only one IF peak was observed at around 536°C during the heating process^[11] clearly differentiated from the 305°C IF peak existing in Fe–Al and pure Al. It is reasonable to conclude that the peaks in the Fe–Al and pure Al powder compact have the same physical mechanism, i.e., originating from the micro-sliding of weak bonding interface between Al powder particles correlated to a recrystallization process of deformed Al particles.^[12]

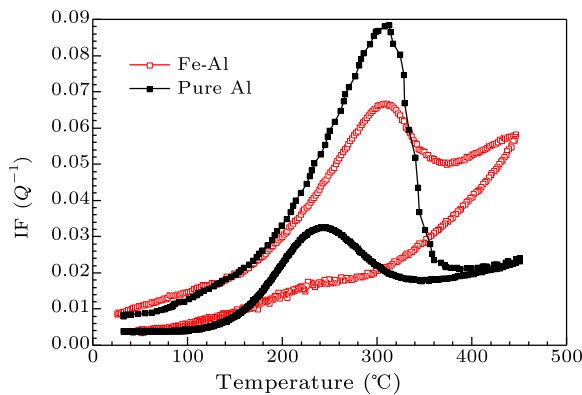


Fig. 4. Comparison of the P_1 internal friction peak for the Fe–Al system and pure Al.

The heat flow effect observed by DSC is shown in Fig. 5 in the temperature range 200–750°C with the heating rate 5°C/min. Two exothermic peaks can be found at around 604°C and 643°C. The first exothermic peak is of a wide distribution range and lower peak height, whereas the second is sharper with much higher peak height. The DSC results are in accordance with the data reported by Charlot,^[5] Sina,^[7] and Hao.^[13] From the profile of the Fig. 5, it can be found that the first peak is more time-dependent which controls the formation mechanism, while the reaction which causes the second peak is more instantaneous. The first exothermic peak was generally recognized to originate from the formation of hard and fragile Fe_2Al_5 , and the nucleation and growth of a secondary FeAl solid solution are responsible for the secondary peak. Compared the DSC properties with the IF features exhibited in Figs. 1–3, it can be found that the temperature locations of P_2 and P_3 IF peaks are quite well consistent with those of two exothermic peaks. It is reasonable to conclude that P_2 and P_3 IF peaks and the DSC peaks are associated with the same physical mechanism.

It is well-known that the Al atom has higher intrinsic diffusion coefficient than that of Fe atoms. In the first stage, the solid diffusion reaction will take place when the temperature reaches about 560°C, i.e.,

$2\text{Fe} + 5\text{Al} \rightarrow \text{Fe}_2\text{Al}_5$. A small amount of Fe_2Al_5 phase generates and disperses along the surface of the Fe particle. As a result, the appearance of the Fe_2Al_5 can be taken into account due to the P_2 IF peak. However, constrained limited contact areas between Fe and Al particles as well as the intrinsic mechanism of solid-solid diffusion reaction, the released heat is smaller and thus the quantity of the Fe_2Al_5 phase formed is limited. Consequently, the weak exothermic peak and lower peak height of the P_2 IF peak are caused. When the temperature arrives at about 645°C corresponding to the second stage of the heating process, a thermal explosion reaction process is ignited and a large amount of heat is emitted, thus instantaneously high temperature comes into being even more than 1000°C.^[14] Much released heat leads to the steep exothermic peaks. The much higher temperature results in the phase transition of Fe_2Al_5 to FeAl, i.e., $\text{Fe}_2\text{Al}_5 + 3\text{Fe} \rightarrow 5\text{FeAl}$. The formation of abundant FeAl phase is responsible for the appearance of the P_3 IF peak.

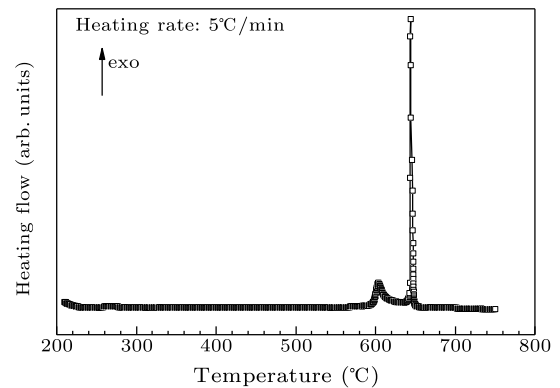


Fig. 5. DSC profile of Fe–Al powder compact.

In summary, internal friction measurement associated with the DSC detection has been used to investigate the phase formation of a Fe–Al powder compact during the heating process. It is found that the internal friction phenomena are very sensitive to response the phase transition process. Three internal friction peaks are observed to appear at different temperature locations. The internal friction peaks exhibit clearly measuring frequency dependence and the heights of the peaks increases as the frequency decrease. Corresponding to the appearance of the second and third internal friction peaks, two exothermic peaks are found in the DSC test and the peak temperatures well match the IF peak temperatures. It is rationalized that the first internal friction is ascribed to the micro-sliding of weak bonding interface associated with a recrystallization process between Al powder particles. The second internal friction peak is associated with the formation of Fe_2Al_5 owing to the solid diffusion reaction at around 560°C. The third internal friction peak at around 640°C arises from the formation of FeAl phase due to the fact that a thermal explosion reac-

tion process near the temperature is ignited and much amount heat is released to induce the phase transition of Fe_2Al_5 to FeAl . In other words, the internal friction peak appearing in the heating process of the Fe–Al powder compact can be recognized as an index to better understand the phase formation and transition.

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