

# Internal friction in advanced Fe–Al intermetallics

J. San Juan<sup>a,\*</sup>, M.L. Nó<sup>b</sup>, J. Lacaze<sup>c</sup>, B. Viguier<sup>c</sup>, D. Fournier<sup>d</sup>

<sup>a</sup> Dpt. Física Materia Condensada, Facultad de Ciencia y Tecnología, Universidad del País Vasco, Apdo. 644, 48080 Bilbao, Spain

<sup>b</sup> Dpt. Física Aplicada II, Facultad de Ciencia y Tecnología, Universidad del País Vasco, Apdo. 644, 48080 Bilbao, Spain

<sup>c</sup> CIRIMAT, ENSIACET, 118 route de Narbonne, F-31077 Toulouse Cedex 4, France

<sup>d</sup> Turbomeca, 64511 Bordes, France

## Abstract

We have measured the internal friction and the dynamic modulus of an Fe–38 at.% Al alloy using a forced torsion pendulum working between  $10^{-3}$  and 10 Hz. The measurements have been carried out as a function of temperature (from room temperature to 1200 K) and as a function of frequency. Two peaks have been observed in the internal friction spectra, at about 780 and 1100 K, which are largely superposed in the intermediate temperature range. Both peaks and the corresponding modulus defect shift in temperature with the oscillation frequency, and can be attributed to relaxation mechanisms. Previous results in the literature seem to indicate that the low temperature peak is the Zener relaxation of Al atoms. The activation energy of the high-temperature peak, referred to as P1 peak because it has not been studied previously, has been determined to be  $H_{\text{act}} = 2.87$  eV, from the results of measurements at different temperatures and frequencies. We discuss the possible mechanisms, which could be responsible of this P1 peak, and suggest that it could be attributed to the intrinsic movements of  $\langle 100 \rangle$  perfect dislocations over the Peierls barrier by kink pair formation in the B2 ordered FeAl.

**Keywords:** Intermetallics; Fe–Al; Internal friction; Peierls barrier; Dislocation relaxation; Zener relaxation

## 1. Introduction

Intermetallic compounds are being developed for high-temperature applications, particularly in the aeronautic industry for high-temperature operating parts in engines, as well as for structural applications at high temperatures in several industrial sectors. This is due to the attractive mechanical behavior: the anomalous (positive) temperature dependence in the yield strength [1,2]. In order to guarantee the performances required in industrial applications, the microstructural stability at high temperatures should be well known. For this purpose, the mobilities of defects and their microscopic mechanisms in the temperature range for the expected application should be studied. Among the different families of intermetallic compounds, the present work focuses on iron aluminides, whose mechanical properties have been overviewed elsewhere [3].

The aim of the present work is to study the mechanisms of relaxation effects due to the mobility of crystal defects in a

polycrystalline Fe–Al intermetallic alloy by mechanical spectroscopy. Anelastic relaxation effects in Fe–Al alloys have been the subject of many papers, and have been recently reviewed [4]. They include the well identified carbon Snoek peak (at around 430 K, for 1 Hz), which is due to short-distance migration of interstitial carbon atoms, and the Zener peak (at around 780 K for 1 Hz), which is due to the stress-induced reorientation of Al atom pairs. Another relaxation, called X relaxation, appearing around 600 K is yet under study [4]. However, no systematic studies have been carried out at temperatures above the Zener peak, viz. above 900 K; and only Wert [5] reported a peak at 1023 K and qualitatively attributed it to grain-boundary relaxation. In the present work, we have measured the internal friction of an Fe–Al alloy in a broad range of temperature between 300 and 1200 K to study the high-temperature relaxation effects.

## 2. Experimental conditions

The samples have been produced by powder metallurgy as an advanced prototype alloy at a laboratory scale, and were subjected to investigations in several laboratories in the framework

\* Corresponding author. Tel.: +34 94 601 2478; fax: +34 94 601 3500.  
E-mail address: [jose.sanjuan@ehu.es](mailto:jose.sanjuan@ehu.es) (J. San Juan).

of a European COST project. The gas atomized powders of Fe–38.8 at.% Al were mixed with 1 vol.% of powders of  $Y_2O_3$ , compacted by hot isostatic pressing (HIP) and hot-extruded in a stainless steel can. From the extruded rod, furnished by Turbomeca, samples of  $50\text{ mm} \times 5\text{ mm} \times 1\text{ mm}$  were cut by electro-sparking in parallel to the extrusion direction. The microstructure shows a distribution of small grains between  $0.5$  and  $10\text{ }\mu\text{m}$  elongated along the extrusion direction, with a very fine dispersion of particles containing Y and Al with a size between  $50$  and  $500\text{ nm}$ , which prevents recrystallization up to temperatures above  $1473\text{ K}$ .

Measurements of internal friction and dynamic modulus have been carried by forced vibration as a function of temperature (between  $300$  and  $1200\text{ K}$ ) and as a function of frequency (between  $10^{-3}$  and  $10\text{ Hz}$ ) in a mechanical spectrometer of a torsion pendulum type described elsewhere [6]. All the measurements have been carried out under a vacuum of  $5 \times 10^{-4}\text{ Pa}$ , and with an oscillation amplitude of  $\varepsilon = 5 \times 10^{-6}$ .

### 3. Results

Fig. 1 shows the internal friction spectrum and the modulus curve for a frequency of  $1\text{ Hz}$ . An internal friction peak is observed at about  $1120\text{ K}$ , which we will call P1, as well as a shoulder between  $750$  and  $900\text{ K}$ . The peak P1 is accompanied with a very large modulus defect. Such a peak has never been reported for Fe–Al alloys. In Fig. 2 we show the internal friction spectra obtained at different frequencies between  $1$  and  $0.003\text{ Hz}$ . The background growing with temperature, which is more clearly visible in the low frequency curves, makes the analysis of the peaks difficult, as we will comment later. The peak P1 is shifted towards low temperature when lowering the frequency, showing the typical behavior of a thermally activated relaxation peak. The peak was also observed in isothermal measurements as a function of frequency and was in fact accompanied with

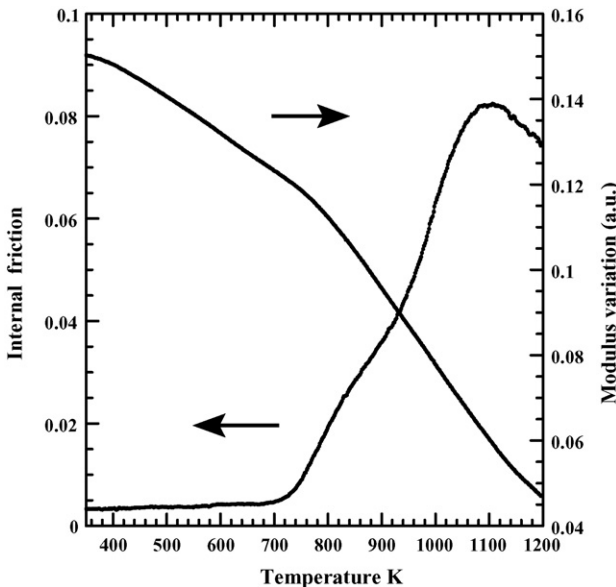


Fig. 1. Internal friction and the variation of the elastic modulus of an Fe–38 at.% Al alloy measured at  $1\text{ Hz}$ .

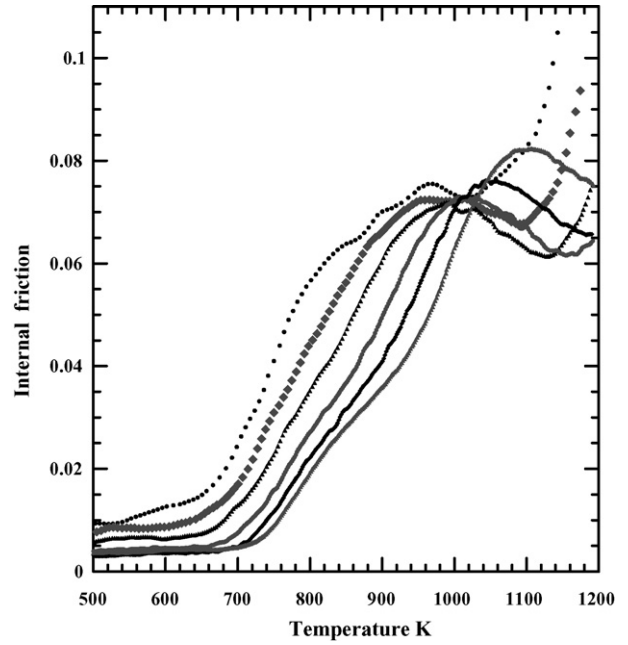


Fig. 2. Internal friction spectra of an Fe–38 at.% Al alloy measured at different frequencies. From right to left, in the middle of the figure:  $1\text{ Hz}$  (grey triangles),  $0.3\text{ Hz}$  (black rhombi),  $0.1\text{ Hz}$  (grey dots),  $0.03\text{ Hz}$  (black triangles),  $0.01\text{ Hz}$  (grey rhombi) and  $0.003\text{ Hz}$  (black circles).

the characteristic modulus defect, as shown in Fig. 3. Fig. 4 presents the internal friction spectra obtained by isothermal measurements at different temperatures between  $900$  and  $1050\text{ K}$ . These results demonstrate a systematic shift in frequency and a decrease of the strength of the peak with increasing frequency at lower temperatures. The latter feature is theoretically expected [7] and widely observed experimentally for Debye relaxation peaks [8].

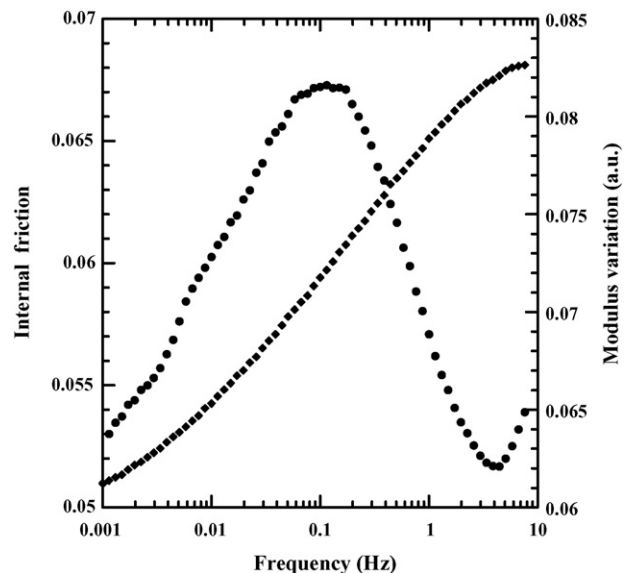


Fig. 3. Internal friction (circles) and elastic modulus (rhombi) of an Fe–38 at.% Al alloy measured as a function of frequency in isothermal condition at  $975\text{ K}$ .

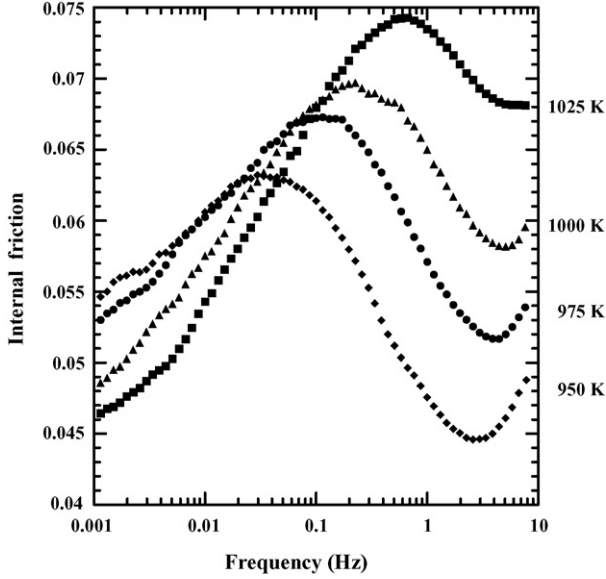


Fig. 4. Internal friction spectra measured as a function of frequency in isothermal conditions at different temperatures as indicated.

#### 4. Analysis and discussion

There is no doubt that the P1 peak is a relaxation peak, because of its behavior in temperature and frequency. However, this peak has never been analysed. We have determined the activation energy from the Arrhenius diagram obtained with the high frequency curves in Fig. 2. This diagram is plotted in Fig. 5, which gives an activation enthalpy of  $H_{act} = 2.85$  eV ( $\pm 0.1$  eV). However, the high-temperature background cannot be subtracted from these curves, and the maximum of the peak is shifted towards high temperature, as much as the peak is at

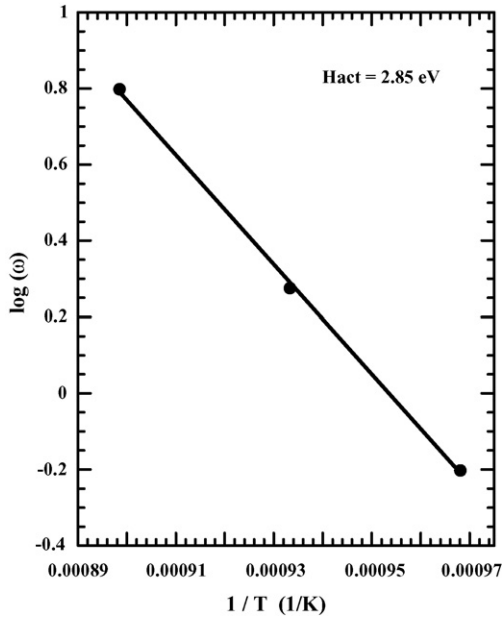


Fig. 5. Arrhenius diagram for the peak position in three of the internal friction spectra shown in Fig. 2, which were measured at different angular frequencies  $\omega$  ( $=2\pi f$ ).

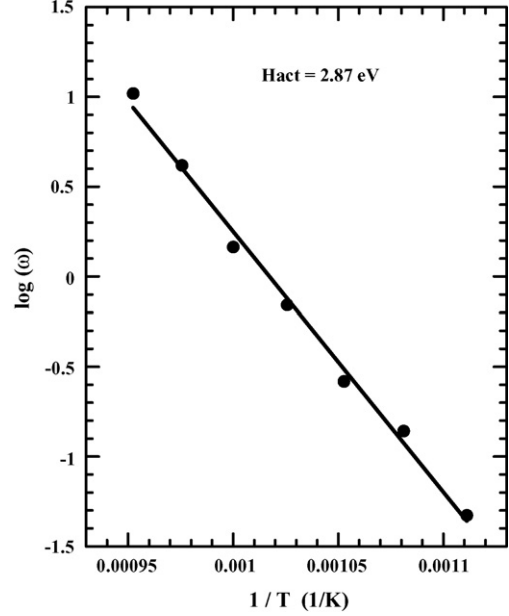


Fig. 6. Arrhenius diagram for the peak position in the complete series of spectra measured at different temperatures as a function of angular frequency  $\omega$  as in Fig. 4.

higher temperature, given place to an underestimation of the activation enthalpy. More reliable determination is made from the spectra as a function of frequency, some of which are shown in Fig. 4. The Arrhenius diagram is presented in Fig. 6, which gives an activation enthalpy of  $H_{act} = 2.87$  eV ( $\pm 0.05$  eV) and a pre-exponential factor of  $\tau_0 = 10^{-15}$  s. Obviously, both diagrams from Figs. 5 and 6 cannot be plotted together because, to do that, one needs to subtract previously the backgrounds to obtain the real absolute temperature of the peaks [9].

In order to interpret the observed internal friction spectra, it is to be noted that the alloy is in the domain of the ordered B2 FeAl phase in the phase diagram [10]. In this phase, the Zener relaxation, which is caused by stress-induced reorientation of solute-atom pairs, has been found around 780 K (for 1 Hz) by several authors, with activation energies of 2.31 eV [11,12], 2.38 eV [13], 2.36 eV [14], 2.35 eV [15], 2.39 eV [4] and 2.46 eV [16]. These values agree with the activation enthalpy for diffusion of vacancies in B2 FeAl, 2.33 eV [17] and 2.48 eV [3]. So, we can attribute the shoulder observed in the internal friction spectra around 780 K (for 1 Hz) to the Zener relaxation, which overlap with the low temperature side of the P1 relaxation.

Concerning the P1 peak, the interpretation is more complex. We have to consider all the possibilities of mobile defects. Nevertheless, we do not need to consider a mechanism linked to the point defect relaxation, like a Zener relaxation, at such high temperatures. Besides, the theoretical analysis of the peak P1, which is out of the scope of the present work, allows us to obtain a broadening of the peak of the Gaussian distribution [18,9] with  $\beta \approx 4$  [19], which is too high for such a kind of relaxation. On other hand, the previous suggestion by Wert [5], a grain-boundary peak, does not seem pertinent in the present case, because the grain boundaries are completely blocked by the  $Y_2O_3$  particles, and other very small particles (less than

50 nm) of Y and Al oxides. In this situation, the P1 peak could be attributed to dislocations.

The previous internal friction studies on Fe–Al alloys were performed mainly on as-cast alloys. The reason why this peak has not been previously observed could be the low dislocation densities in as-cast alloys. Attributing the P1 peak to dislocations is consistent with previous works by electron microscopy and plastic deformation on Fe–Al alloys of similar compositions [3,20], which showed that, above 773–873 K,  $\langle 100 \rangle$  perfect dislocations control deformation. These dislocations are formed by the decomposition of  $\langle 111 \rangle$  dislocations, which control the deformation at low temperatures [21]. If such dislocations are not glissile at low temperatures, the first step of the mechanism should be to overcome the Peierls barrier. The observed P1 relaxation peak could thus be attributed to the kink pair formation (KPF) mechanism of the  $\langle 100 \rangle$  dislocations on their  $\{100\}$  glide planes, as the classical Bordoni peak in pure metals, but in non-compact planes. Besides, the measured  $\tau_0$  for the P1 peak,  $10^{-15}$  s, corresponds well to a dislocation motion controlled by the KPF mechanism: in bcc metals the value of  $\tau_0$  for the  $\gamma$  peak due to KPF on screw dislocations is between  $10^{-13}$  and  $5 \times 10^{-17}$  s [22], and in particular it is in between  $4 \times 10^{-13}$  and  $1.2 \times 10^{-15}$  s for pure iron [23].

The proposed interpretation is in accordance with the observations by transmission electron microscopy, in which  $\langle 100 \rangle$  dislocations, just after the indicated reaction at about 780 K, were observed to remain fairly straight and rather immobile at this temperature, presumably because of the Peierls friction forces [20]. Indeed, this temperature is at the beginning of the observed P1 relaxation peak, which would be linked to the intrinsic motion of such  $\langle 100 \rangle$  dislocations in the B2 ordered phase. This interpretation has an obvious interesting consequence, because this relaxation peak is then directly relevant to the fall of the yield stress anomaly observed in Fe–Al intermetallic alloys [3].

### Acknowledgments

The authors thank the Conseil Général Midi-Pyrénées (project no. 0300752) and the Departamento de Educación

of the Basque Government (project CTP-P9) for the financial support.

### References

- [1] N.S. Stoloff, V.K. Sikka (Eds.), *Physical Metallurgy and Processing of Intermetallic Compounds*, Chapman and Hall, New York, USA, 1996.
- [2] J.H. Westbrook, R.L. Fleischer (Eds.), *Intermetallic Compounds*, John Wiley & Sons, Chichester, UK, 2000.
- [3] I. Baker, P.R. Munroe, *Int. Mater. Rev.* 42 (1997) 181–220.
- [4] I.S. Golovin, H. Neuhauser, A. Riviere, A. Strahl, *Intermetallics* 12 (2004) 125–150.
- [5] C. Wert, *J. Appl. Phys.* 26 (1955) 640–641.
- [6] I. Gutierrez-Urrutia, M.L. Nó, E. Carreño-Morelli, B. Guisolan, R. Schaller, J. San Juan, *Mater. Sci. Eng. A* 370 (2004) 435–439.
- [7] G. Fantozzi, in: R. Schaller, G. Fantozzi, G. Gremaud (Eds.), *Mechanical Spectroscopy Q-1 2001*, Trans Tech Publications, Uetikon, Switzerland, 2001, pp. 3–31.
- [8] A. Riviere, P. Gadaud, *J. Phys. IV* (1996) C8–C81.
- [9] J. San Juan, in: R. Schaller, G. Fantozzi, G. Gremaud (Eds.), *Mechanical Spectroscopy Q-1 2001*, Trans Tech Publications, Uetikon, Switzerland, 2001, pp. 32–73.
- [10] O. Kubaschewski, *Iron-binary phase diagrams*, Springer-Verlag, Berlin, Germany, 1982, pp. 5–9.
- [11] K. Tanaka, *J. Phys. Soc. Jpn.* 30 (1971) 404–411.
- [12] K. Tanaka, K. Sahashi, *Trans. JIM* 3 (1971) 130–135.
- [13] W. Hermann, T. Ort, H.G. Sockel, in: M.V. Nathal, R. Darolia (Eds.), *Structural Intermetallics 1997*, TMS, Warrendale, USA, 1997, pp. 759–768.
- [14] N.Y. Rokhmanov, *Funct. Mater.* 7 (2000) 235–239.
- [15] I.S. Golovin, T.V. Pozdova, R.V. Zharkov, S.A. Golovin, *Mater. Sci. Heat Treat. Metals* 6 (2002) 16–22.
- [16] Z. Zhou, Z. Gao, F. Han, *J. Phys.: Condens. Matter* 15 (2003) 6809–6815.
- [17] J. Wolff, M. Franz, A. Broska, B. Kohler, Th. Hehenkamp, in: M.V. Nathal, R. Darolia (Eds.), *Structural Intermetallics 1997*, TMS, Warrendale, USA, 1997, pp. 721–725.
- [18] A.S. Nowick, B.S. Berry, *Anelastic Relaxation in Crystalline Solids*, Academic Press, New York, USA, 1972.
- [19] J. San Juan, M.L. Nó, in: P. Maugeis, J. Lacaze (Eds.), *Discussion Meeting on the Development of Innovative Iron Aluminium Alloys*, Toulouse, France, 2005.
- [20] D.G. Morris, M.A. Morris, *Mater. Sci. Eng. A* 239–240 (1997) 23–38.
- [21] P.R. Munroe, I. Baker, *Acta Metall. Mater.* 39 (1991) 1011–1017.
- [22] I.G. Ritchie, G. Fantozzi, in: F.R.N. Nabarro, (Ed.), *Dislocations in Solids*, vol. 9, 1992, pp. 57–133.
- [23] H. Mizubayashi, H. Kronmuller, A. Seeger, *J. Phys. (Paris)* 46 (1985) C10–C309.