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Calorimetry study of $Ni_{50}Ti_{50}$ ball milled powders

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Abstract

The variation of crystallization temperature T_x and activation energy E_x with ball milling time in mechanical alloying were investigated for $Ni_{50}Ti_{50}$ mixture powders. It was found that T_x and E_x for a milling time of 20 h are higher than the documented values and decrease rapidly with increasing milling time.

1. Introduction

The preparation of amorphous alloys by solid state reaction (SSR) and in particular the synthesis of amorphous alloy powder by mechanical alloying (MA) are now the subject of increasing interest. However, many aspects of these processes and the basic features of the transformation are still unclear and debatable, although much attention has been paid to characterization of these metastable materials. Obviously, depending on the experimental conditions adopted, both the process route and the products will differ from one case to another.

In this paper we report differential scanning calorimetry (DSC) analysis data of $Ni_{50}Ti_{50}$ powder prepared by ball milling for various lengths of time, and present a comparison with another work published in the literature on a similar system.

2. Experimental details

$Ni_{50}Ti_{50}$ amorphous alloy powders were prepared by mechanical alloying of mixtures of powders of nickel and titanium under a pure argon atmosphere with a high energy ball mill. The vial used was made of stainless steel and the ball was made of WC. Samples were taken out of the vial after selected milling times for X-ray diffraction and DSC measurements. For DSC measurements, the powder sample was placed in an aluminium pan and the cell was evacuated before the runs, and then pure argon obtained by purification equipment was employed as a purging gas at a rate of 40–120 ml min^{-1}.

3. Results and comparisons

Figure 1 shows selected X-ray diffraction patterns of the $Ni_{50}Ti_{50}$ powder milled for a given time. We can see from this figure that the intensity of

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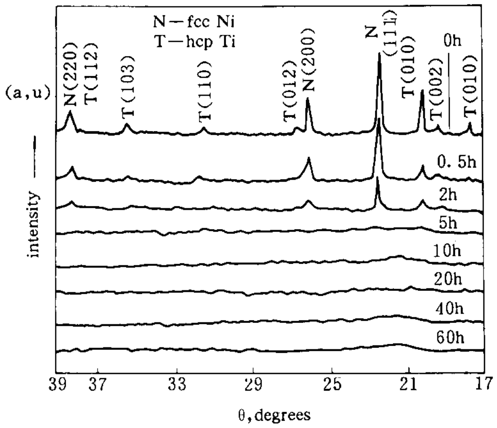
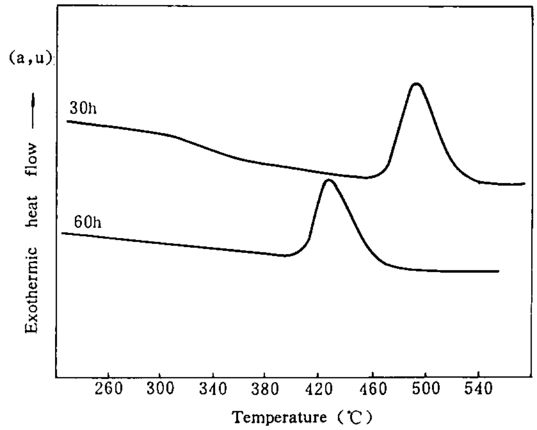


Fig. 1. X-ray diffraction diagrams for the $Ni_{50}Ti_{50}$ system at the indicated milling times.



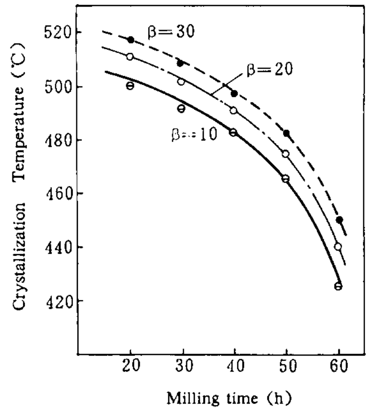


Fig. 2. DSC trace of $Ni_{50}Ti_{50}$ ball-milled powders. The heating rate was 10 K min^{-1}.

Fig. 3. The peak temperature of crystallization T_p as a function of ball-milling time. β (C min^{-1}) is the heating rate.

the diffraction peaks related to h.c.p. titanium and f.c.c. nickel crystalline phases decreases gradually as the milling time increases. After the powders were milled for 5 h, all the peaks of crystalline phases had disappeared.

The DSC measurements show that any exothermal or endothermic peaks do not emerge before a milling time of 10 h, in spite of the fact that there were no diffraction peaks in the X-ray pattern after a milling time of 5 h. An exothermic peak only appears after a milling time of 20 h in DSC traces. Figure 2 gives the DSC traces of powders milled for 30 h and 60 h. It is shown in the figure that there is only one exothermal peak in the DSC trace, being dissimilar to that given in refs. 1 and 2. The crystallization enthalpy ΔH_x of the powders prepared by MA for 20–60 h is approximately 1.5 kJ mol^{-1}.

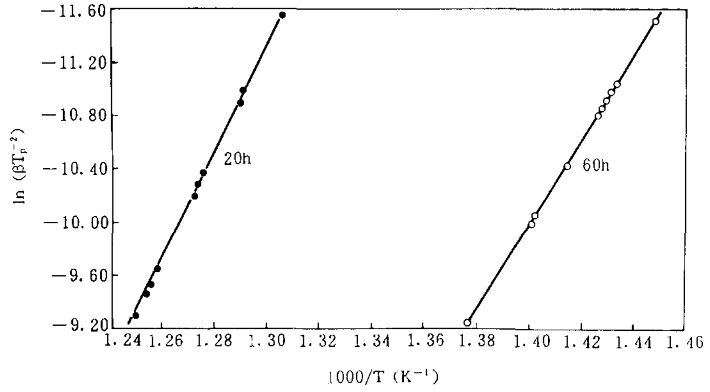


Fig. 4. The activation energy for crystallization determined by the Kissinger peak shift method.

The variation of crystallization temperature for MA powders with milling time is given in Fig. 3. It is shown that the crystallization temperatures T_x are higher than those given in other work [1], furthermore they decrease rapidly with increasing milling time, which differs remarkably from the results in ref. 3. In view of the results shown in Fig. 3 of ref. 4 that T_x has a minimum near the composition of the intermetallics Ni_3Ti and $NiTi_2$ and a maximum near NiTi, the difference can hardly be explained by a composition change along with the milling process. It can only be ascribed to a structural change in the amorphous products themselves.

The activation energy E_x for the crystallization of the MA products corresponding to milling times of 20 h and 60 h was determined by the Kissinger's peak shift method. According to Fig. 4, we obtained the energy E_x as 336 kJ mol^{-1} for 20 h and 269 kJ mol^{-1} for 60 h. It is shown that the activation energy decreases with milling time, but the values are higher than those given in ref. 2.

Acknowledgment

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