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Preparation of nanocrystalline intermetallic compounds WSi_2 and $MoSi_2$ by mechanical alloying

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Abstract

Nanocrystalline intermetallic compounds WSi_2 and $MoSi_2$, with grain sizes of about 14 nm and 43 nm respectively, were synthesized by mechanical alloying from elemental powders of W or Mo and Si. The microstructure development of the powder mixtures during mechanical alloying was monitored by X-ray diffraction experiments. After milling for 30 h, a solid-state reaction between W and Si or Mo and Si occurred with the formation of WSi_2 or $MoSi_2$. When the milling time reached 210 h, these reactions were almost complete.

Keywords: Mechanical alloying; WSi_2; $MoSi_2$

1. Introduction

Since the pioneering work of Gleiter et al. [1], nanocrystalline materials have attracted great attention in the past few years. In the last decade, mechanical alloying proved to be an effective method for the preparation of nanocrystalline materials [2,3], especially for those of intermetallic compounds with high melting points [4,5]. Recently, refractory metal silicides have attracted considerable attention because of their thermal stability. Both WSi_2 and $MoSi_2$ are usually considered difficult to prepare by conventional metallurgical techniques because of their high melting point. In this paper, nanocrystalline intermetallic compounds WSi_2 and $MoSi_2$ are prepared by mechanical alloying and the microstructure development monitored by X-ray diffraction (XRD) experiments. The as-prepared WSi_2 nanocrystalline powder was also analyzed by a differential scanning calorimetric (DSC) experiment.

2. Experimental details

Pure elemental tungsten, molybdenum and silicon powders with particle sizes smaller than 75 μm were mixed with the molar ratio of W:Si or Mo:Si being equal to 1:2. The powder mixtures were put into sealed cylindrical steel vials together with tungsten carbide balls (12 mm in diameter) under an argon atmosphere. The weight ratio of the balls to the powder mixture was 15:1.

Mechanical alloying was performed using a planetary ball mill (QM-F1) at ambient temperature with the planetary rotation speed of 220–240 rpm. At selected times, a small amount of the sample powders were removed for structure analyses. XRD experiments were carried out on a Philips PW 1700 diffractometer with Cu Kα radiation. Thermal analysis was performed on a Perkin–Elmer differential scanning calorimeter at a scan rate of 20 K min^{-1}.

3. Results and discussion

Fig. 1 shows the variation of XRD patterns of the powder mixtures after different milling times. It can be seen that the intensity of Bragg peaks of W (see Fig. 1(a)) or Mo (see Fig. 1(b)) decreased gradually with an increase of milling time. Si diffraction peaks decreased significantly after 10 h milling, and fully disappeared after 30 h milling. Meanwhile, Bragg peaks corresponding to intermetallic compound WSi_2 or $MoSi_2$ emerged. After 210 h milling, the powder mixtures had transformed to WSi_2 or $MoSi_2$ completely, and with further milling no evident change of XRD patterns

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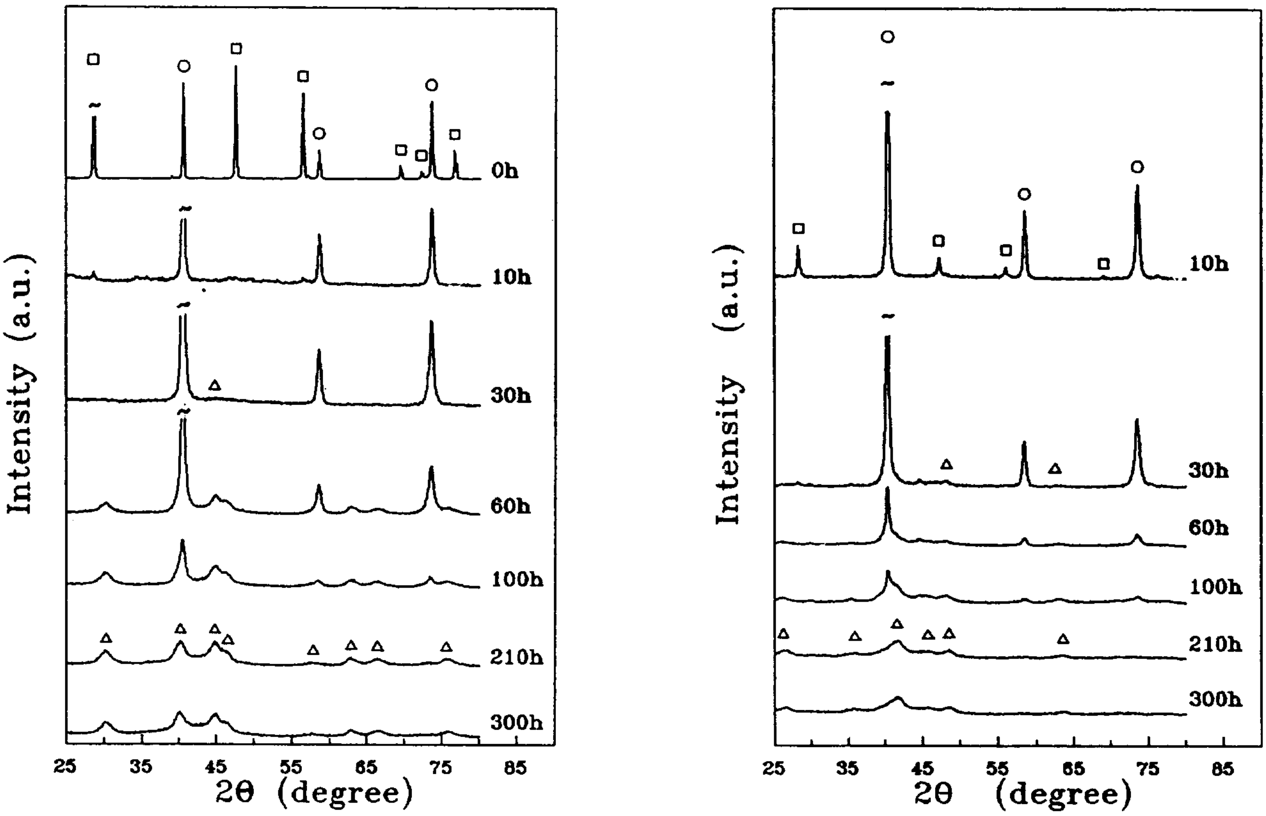

Fig. 1. XRD patterns of the powder mixtures after different milling times. (a) W-Si system: ○, W; □, Si; △, WSi_2. (b) Mo-Si system: ○, Mo; □, Si; △, $MoSi_2$.

was observed. The average grain size of WSi_2 and $MoSi_2$ in the 300 h milled samples were measured by the XRD line profile analysis to be 14 nm with a strain of 2.3% and 43 nm with a strain of 2.6% respectively. It should be note that the strain in the products is very large.

In order to further examine the characterization of the intermetallic compound WSi_2, DSC experiments were performed on WSi_2 after 300 h milling (Fig. 2). It was found that in the temperature ranges 393-533 K and 535-893 K, there are an exothermic process and an endothermic process respectively. Both of them are very broad. The exothermic one may be attributed to lattice relaxation and releasing of storage stress, which was caused by the strong deformation during mechanical alloying. The endothermal process may result from the releasing of adsorbed gas on the large surface area of this nanocrystalline sample.

It is known that during mechanical alloying, the mixture powders are trapped by the colliding balls, heavily deformed, repeatedly flattened and cold welded. The grain size of the original powder gradually decreases and the interface between different components increases after milling. On these interfaces, an intermetallic compound WSi_2 or $MoSi_2$ could be formed, which is a common phenomenon of a solid-state reaction driven by mechanical alloying. This involves two processes: nucleation at the interface, and growth of the nuclei. Both of them are governed by the interdiffusion reaction [6], similar to the solid-state reactions occurring in a multilayer. In addition, the high density of defects and the interface generated by mechanical alloying would promote the reaction process.

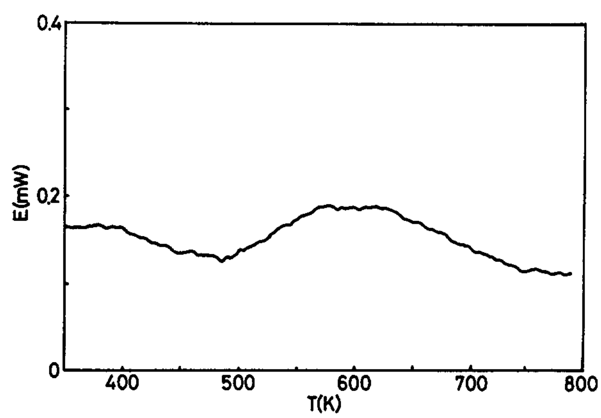

Fig. 2. DSC trace of the as-prepared WSi_2 powder after milling for 300 h.

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