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Effect of mechanical alloying on the structure and properties of $NbCr_2$ Laves phase fabricated by hot pressing

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Abstract

$NbCr_2$ Laves phase intermetallics has been successfully made with mechanically alloyed powders with nominal compositions Cr—33.3 at.% Nb by hot pressing (HP). The effect of mechanical alloying (MA) on the structure and the properties of hot-pressed alloy has been investigated by X-ray diffraction, scanning electron microscopy, and bending test, etc. The results show that the $NbCr_2$ Laves phase prepared from milled powders exhibits high strength and low density in comparison with one prepared from elemental powders. The high density of defects brought during MA and deficiently eliminated by following HP led to the high hardness and low density. Fine grains and dispersion of fine precipitation caused by MA are related to the improvement of strength of alloy.

Keywords: Intermetallics; Powder metallurgy; Scanning electron microscopy; Mechanical alloying

1. Introduction

The Laves phase $NbCr_2$ alloy has a great potential for high temperature structural applications due to its high-melting temperature, appreciable creep resistance, relatively low density (7.7 g/cm^3), and high strength [1–5]. There are three common polytypes of Laves phase $NbCr_2$ most frequently observed: cubic C15 ($MgCu_2$-type) structure below ~1873 K, hexagonal C14 ($MgZn_2$-type) structure between 1873 K and its melting point (between 2003 and 2043 K) and dihexagonal C36 [1,5]. Although there has been a wide research activity in intermetallics in the last years, commercial utilization of $NbCr_2$ was hampered because it is very brittle at low temperature, like other intermetallics.

Mechanical alloying (MA) has shown to be a promising synthesis technique for materials with high-melting temperature [6]. MA is a high-energy ball milling technique for production of microscopically homogeneous materials starting from blended elemental powders. Many high temperature melting intermetallics that are difficult to prepare by conventional processing techniques could be easily synthesized with better mechanical properties by the MA process followed by hot pressing (HP). In this method, it is easy to control the microstructure to enhance the ductility of Laves phase $NbCr_2$. There are a large number of reports on behavior of conventionally cast Laves phase $NbCr_2$ in the past decades, but few studies have been carried out on MA and HP materials consolidated.

The investigations of MA on Laves phase $NbCr_2$ from pre-alloyed and elemental mixture powders have also been carried out [7,8], however, there are few reports on the effects of mechanical alloying on the structure and properties of densified materials.

In this paper, the fabrication of Laves phase $NbCr_2$ by hot pressing process from mechanical alloyed powders and elemental powders has been investigated. The effects of mechanical alloying on the structure and the properties of hot-pressed alloy have also been studied.

2. Experimental procedures

High-purity crystalline powders of Nb (99.9%, −200 mesh) and Cr (99.9%, −200 mesh) in a molar ratio of 1:2 (i.e., Nb47.1Cr52.9) were milled in vacuum for periods up to 20 h. The milling was carried out in a planetary ball-mill (QM-1SP) with both vial and balls made of hardened steel. To reduce oxidation of

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powders during mechanical alloying, the mechanical alloying was performed in vacuum. Milling parameters such as ball-to-powder mass ratio (13:1) and speed rotation (400 rpm) were kept constant throughout the experiments. During the milling, the powders are liable to adhere to the wall of the pot due to the so-called cold welding effect, causing the decreased milling efficiency or even uncompleted milling. The pot was therefore taken into a glove box every 5 h to remove the adherent powders from the pot wall and to break the agglomerated powders. The powders were taken in a graphite mould with 14 mm diameter and hot-pressed under 45 MPa for 0.5 h at 1523 K in conditions of vacuum.

The samples were characterized by X-ray diffraction (XRD) performed at room temperature, with a D8/ADVANCE X-ray diffractometer using Cu Kα radiation ($\lambda = 0.154056$ nm). The angular scanning was comprised in $20° \leq 2\theta \leq 80°$ range. XRD data was collected at a slow scan rate of 0.005 deg/s for the careful determination of average crystallite size and internal strain using Williamson–Hall method [9] and the method suggested by Ungar et al. [10,11]. Scanning electron microscopy (SEM), using a QUANTA200 microscope, was also performed in powders and the hot-pressed samples. Chemical analysis was conducted to measure oxygen content of as-milled powders. The densities of hot-pressed samples have been determined by buoyancy method applying the Archimedes Principle. Bending specimens with 6 mm in gauge length, 4 mm in diameter were spark cut from the sample. Bending tests were done at a constant strain rate of 0.01/s at room temperature. Microhardness tests were carried out at a load of 1000 g.

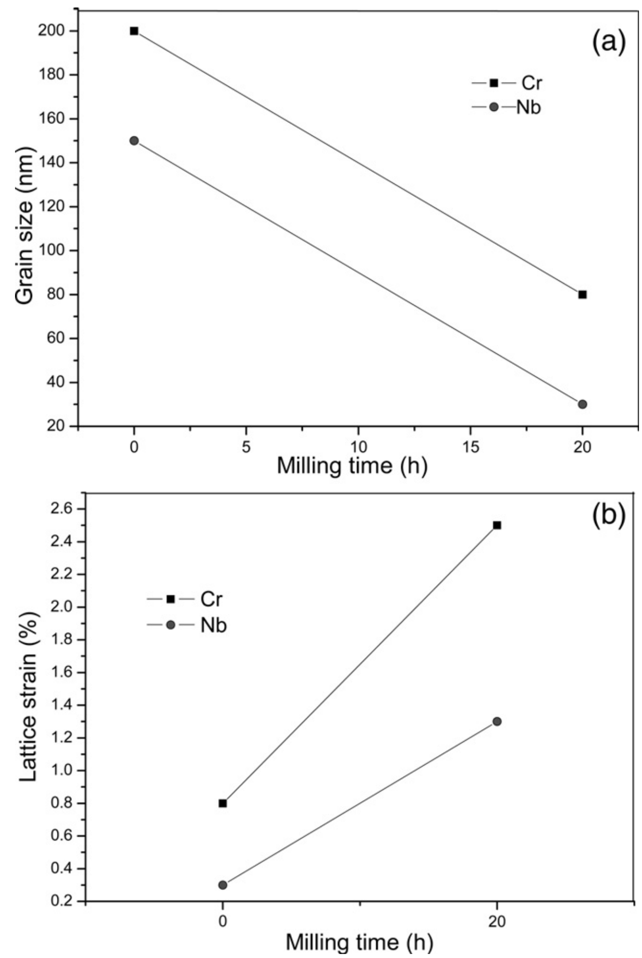

Fig. 2. Variation of grain size (a) and lattice strain (b) with MA time.

3. Results and discussions

3.1. Analysis of powders

The diffraction patterns of elemental mixture powders and as-milled powders are shown in Fig. 1. At the early stage of milling, crystalline Nb and Cr peaks were observed. It can be seen that mechanical alloying leads to significant broadening of X-ray diffraction peaks after 20 h milling, indicating the formation of fine grain and a high density of defects caused by large local strains in the powder particles [as shown in Fig. 2(a) and (b)]. The broadening of X-ray diffraction peaks is associated with the refinement of grain size. At the very beginning of milling, MA leads to a fast decrease of the grain size.

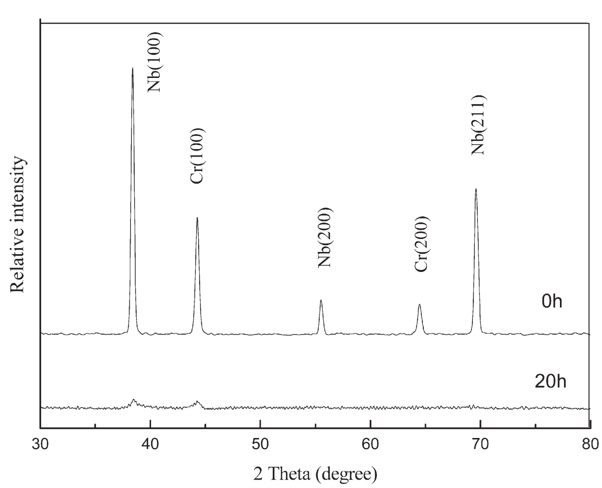

Fig. 1. X-ray diffraction patterns of elemental mixture powders and as-milled powders.

Because a part of Nb atoms dissolved into the Cr lattices, the peaks of Cr were lightly shifted to low angle (the lattice constants changed from 2.8845 Å to 2.8901 Å, calculated from XRD results) and the Nb peaks were weakened. It agrees with the previous results that supersaturated solution Cr (Nb) had been formed by mechanical alloying [7].

The SEM micrograph of elemental mixture powders is shown in Fig. 3(a). The powders have irregular shapes and various sizes. The mean size of particles is about 20 μm. Fig. 3(b) shows the micrograph of as-milled powders. It can be seen that the size of particles is about 3 μm, which tend to be uniform. After 20 h milling, the particles are refined, but some of particles appear slice. During the ball milling, the Nb particles were collided into slices due to their excellent ductility. The Nb slices gradually turn large through welding fine Cr particles around them. Though refining action occurs simultaneously, the welding plays a dominant role in this procedure. The milled powders are more homogenous than elemental powders from the EDX analysis. The distributions of elements of Nb and Cr are symmetrical for all milled powders.