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Fabrication of Equally Axial Channel Extrusion Device for Processing of TiCoSb-based Intermetallics

by

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Abstract

Equally axial channel extrusion (EACE) belongs to the family of severe plastic deformation processing methods, which have the ability to produce ultra-fine grain materials or nano-materials with relative low costs. After summarizing the processing methods for nano-crystalline materials, this paper describes the fabrication of such a device. Preliminary results and characterization of TiCoSb-specimens are described. This research aims to produce thermoelectric materials, and the proposed materials processing method is considered to be environmental friendly because of low costs.

Keywords: Material processing, Nano technology, Thermoelectric materials.

1. Introduction

Equally axial channel extrusion (EACE) is a relatively new processing method for metals or intermetallic alloys [1,2] developed in the last decade. This method, also called equally axial channel pressing (EACP) due to the high applied pressure of powder compaction with subsequent severe plastic deformation (SPD), mainly due to large shearing stresses in a 900 bent pipe. During extrusion, it is important to apply a back stress in order to avoid inhomogenities [3]. Also when the extrusion is applied several times in different directions, certain textures can be adjusted [4]. The main advantage of ECAP is that the heavily worked microstructures typically show high strength, in combination with good ductility at relatively low costs for equipment or process energy. Mechanical properties get improved, even fatigue life becomes longer [5] or low temperature superplasticity occurs [6].The reason is that during SPD the grain get sheared, partly recrystallize and result in Ultra-fine-grain (UFG) or even nano-crystalline (NC) materials with all its micro-structural advantages [7].

There are several processing methods for nano-crystalline materials, the main methods are summarized in Fig. 1. The first nano- crystalline material was produced by condensation of metallic vapor into nano-powder with subsequent sintering [7-10] (Fig. 1f). Rapid quenching of

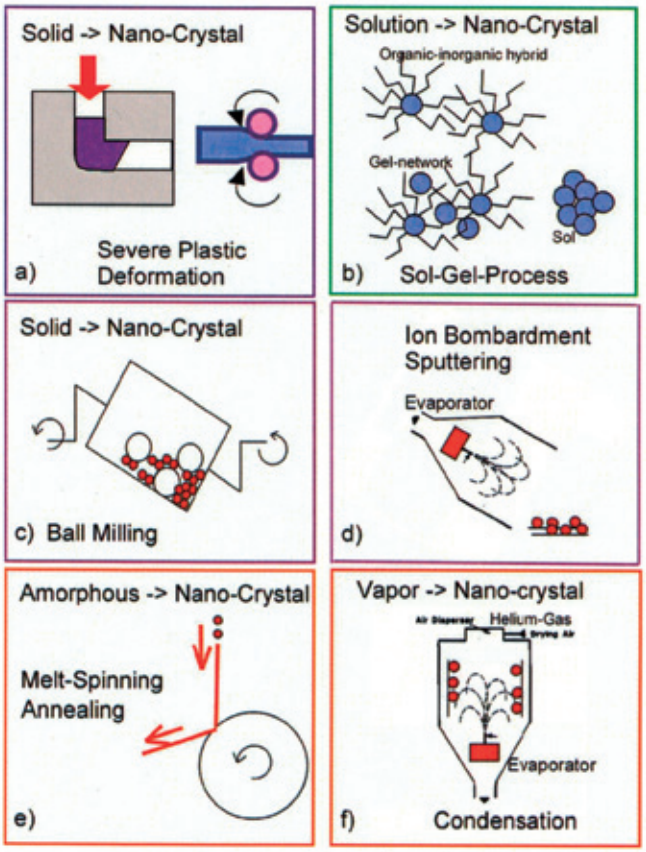

Fig. 1. Six main processing techniques for nanocrystalline materials

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metallic melts leads to metallic glasses [11], which crystallize into nano-materials during annealing (Fig. 1e). Ball-milling [12] and ion-bombardment destroys the crystal lattice on the surface and lead to nano-crystalline re-arrangement (fig. 1 c, d). In chemistry the most often applied method is the sol-gel method, where organic precursor molecules are polymerized into ceramics or ceramic-metal composites [13] (fig. 1 b). The goal of our laboratory is to produce intermetallic alloys for thermoelectric applications [14]. The processing method used until now, requires at least three steps, pressing the powder into pellets, arc-melting and annealing, which should be longer than 100h according to literature [15].

The intention of this paper is to describe the fabrication of a new EACE-equipment in order to produce TiCoSb-based intermetallics in an economic way. The first chapter describes the design of the die, the second chapter the experiments and the last chapter the resolved material.

2. Experimental equipment

In order to fabricate a die for EACP in a 50x70x80mm steel block (SK105) a vertical hole (Φ = 6mm) was cut, and from the top another hole (Φ = 6mm) was drilled until it reaches the first hole, as shown in fig, 2a. This hole is used to fill in the powder and the other hole is used to extrude the produced specimen using as shown in fig, 2a. The plunger is fixed during experiment, but can be released in order to draw the specimen out. A heater (Sakaguchi MS1000) with 25x25x1.5mm dimension and a power of approximately 1000W is applied from the bottom in order to heat the shear chamber. The heater was inserted in a slit produced by rotary grinding. Cooling water inlet and outlet was drilled as holes (Φ = 8mm) and the holes were connected with bended Cu-pipes screwed into the block. Fig. 2b shows the die as manufactured in this stage.

During extrusion experiments it was detected that the power from one heater was not sufficient. For this reason the steel block was rotary-grinded in order to apply three more additional heaters, which were embedded in Isolite fire bricke as shown in fig, 3a. Also, another advantage was to reduce the mass and therefore the heat capacity of the steel block in order to increase the temperature of the device. For applying the back stress a spring was attached at the plunger at the specimen outlet. The spring had a spring modulus according

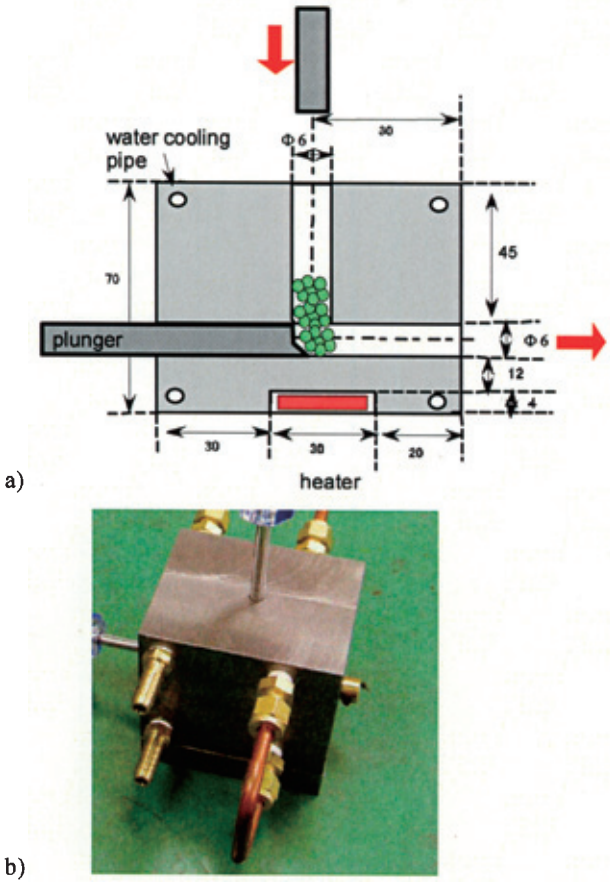

Fig. 2. a) Sketch and b) picture of the main device for equally axial channel extrusion. The left plunger is fixed; the upper plunger applies pressure to the powder which is heated from the bottom and both sides. During experiment a third plunger is attached from the right, which applies the back stress.

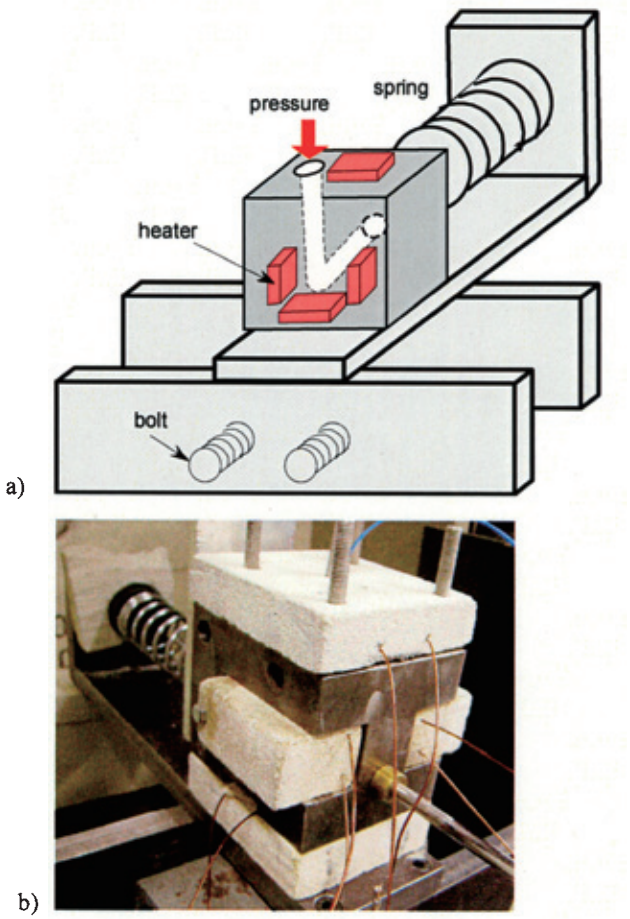

Fig. 3 a) Sketch, and b) preliminary realization of the attachment of the EACE-device to the deformation machine. The spring in the rear part applies back stress to the specimen.

to Hook's law of 0.0036 N/mm as measured in a compression experiment. With the plunger diameter of Φ = 6mm a stress of 159 MPa is applied in the case of a 15mm long specimen and 319 MPa in the case of 20mm length. The die was attached to an Instron deformation test machine usually used for tensile tests. This machine applies a sufficiently high compressive force (4.4 kN) when attached in reverse direction. For the die this yields to a compression stress of more than 10 GPa.

The design of the attachment is shown in fig. 3a using two bolts in order to connect the die with the deformation machine. In a preliminary realization (as shown in fig, 3b), the connection is performed by several 1mm steel wires. The connection is necessary in order to remove the upper plunger after the experiment against friction.

The temperature is measured by thermocouples attached to Sanwa PC 510 voltmeters. One thermocouple is attached directly to one of the heaters in order to measure the input heat, another is applied to a hole near the plunger It was found that the die has almost constant temperature at any place during heating due to its high thermal conductivity; only the surface is about 100 K lower. The data were recorder via an USB link to a computer and stored in-situ.

3. Experimental procedure

Ti, Co and Sb powders with defined weight ratios were filled into the tube and a pressure of 10 MPa applied. Then the heaters were turned on slowly. All heaters are form the same type and they were heated with same electric power. When increasing the voltage, the temperature increases as shown in fig. 4. Due to the large mass of the die, it takes time of about 20 min in order to achieve equilibrium. The increase of the temperature is proportional to the input voltage and hence the input power, because the resistance does not change much with temperature. This is confirmed by the straight line above

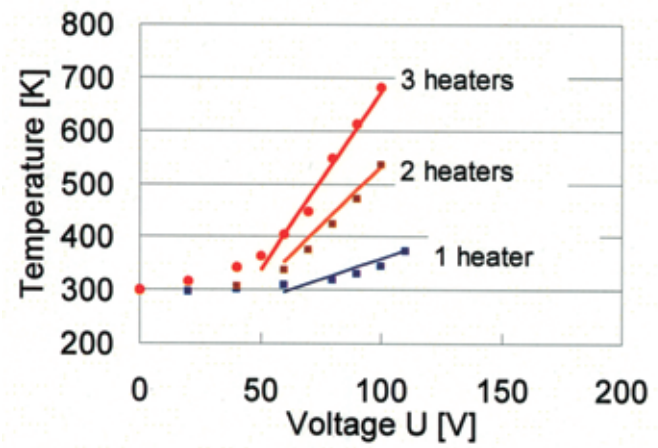

Fig. 4 Measured temperatures during heating. The lines are guides to the eye.

about 400K. When the temperature is lower, it takes longer time to reach equilibrium. This explains the deviations.

During heating the applied compression stress reduced to about 7 MPa. When the maximum temperature is reached, the compression stress was increased. The powder particles get sheared and due to temperature they react with each other. After the experiment the air cooling needs about three hours. When water cooling is used, the cooling time reduces remarkably.

4. Result and discussion

The reacted specimens were analyzed by SEM. Compared with arc-melted specimen with a grain size of about 15 μm shown in fig. 5a, the grain size is much smaller (about 1.5 μm). The composition has not yet been analyzed, but the

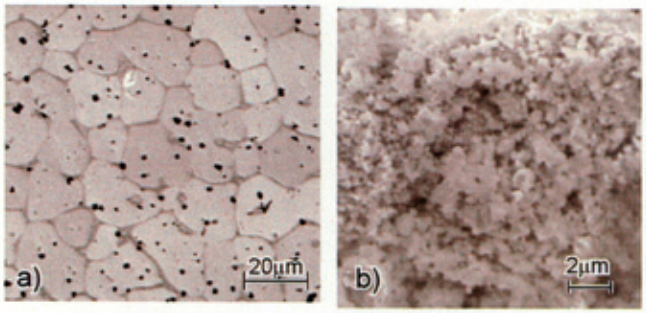

Fig. 5 TiCoSb specimen produced by a) arc-metlting, b) the EACE device

desired formation of the half-Heusler phase TiCoSb is expected. Compared to the arc melting and subsequent annealing the specimens can be produced much faster and with less amount of energy, Hence, this method is also concerned as environmental friendly processing technique. A further increase in processing temperature from presently 700 K to about 900 K is desired in order to make processing easier and smoother. However, when this equipment is used for student's education, special care is necessary for safety reasons.

5. Conclusion

This is the first and preliminary report of manufacturing an ECAE device. Specimens can be produced faster and with less amount of energy than previously used techniques. This technique has the ability to produce compact composite materials with nano-scale dimensions.

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