SEMI-SOLID PROCESSING OF METAL-OXIDE COMPOSITE

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Abstract

One of the available routes to developing a new material resistant to high-temperature creep is to create a microstructure consisting of a metal matrix and dispersed stable particles. For making intricately shaped components from such materials, new processes must be found to allow near net shape products to be manufactured in a simple and rapid manner. A semi-solid processing chain relying on mini-thixoforming could become one such process. For this purpose, an unconventional technology chain was designed in the present experiment. The chain comprises mechanical alloying, powder metallurgy techniques and thermomechanical treatment with transition through the semi-solid state. In this chain, thanks to the intensive deformation at the thixo-forming stage, the desired shape is achieved effectively. The second requirement was the good creep resistance of the material. To this purpose, two different powder materials consisting of metals and oxides were proposed. In both cases, the metal constituent contained iron and aluminium. The primary difference between the materials denoted as A and B was the nature of the oxides acting as strengthening particles. The powder mixture was prepared by mechanical alloying and compacted using various techniques. The powder mixture for the A material was compacted using high-pressure torsion (HPT). The B material was compacted by heating the mechanically alloyed powder enclosed in a steel container in a furnace and by subsequent intensive compressive deformation in a press. From the resulting products, cylindrical specimens for semi-solid processing were cut. The rate of heating to the semi-solid processing region was high. In the first stage, appropriate temperatures and heating rates were sought and optimized. These are crucial in obtaining the desired fine and adequately uniform dispersion of particles providing the strengthening effect. Once solidified and cooled, the materials exhibited dense structures free of pores, with uniformly distributed particles. The hardness values for the A material were close to 460 HV5, the hardness range of the B material was 360 - 447 HV10 and the values varied, depending on the processing parameters.

Keywords: Metal-oxide composite, high temperature creep, mechanical alloying, mini-thixoforming

1. INTRODUCTION

A combination of powder metallurgy and semi-solid processing techniques, namely thixo-forming, may provide new opportunities in manufacturing complex-shaped components with high creep resistance. Small parts with a volume of up to 1 cm^3 may be produced by means of mini-thixoforming. With this technology, the feedstock can be heated within very short times in the order of tens of seconds. Using an alloy with finely dispersed oxide, excellent properties, namely creep resistance, can be achieved at significantly reduced input material costs when compared to the currently used superalloys. Non-conventional semi-solid processing (i.e. processing in the freezing range) of mechanically alloyed powder mixtures in a single technology chain offers potential for effective manufacturing of intricate-shaped parts [1-2].

The difficulty in processing powder mixtures lies in the various melting points of the various constituents and in identifying the appropriate processing temperature. The choice of powder preparation techniques, heating rates and holding times, the magnitude, rate and other aspects of the applied deformation is therefore the key to the successful implementation of such a process [3, 4]. Besides producing the desired shape of the part, it

is essential to keep the strengthening oxide particles as small and stable as possible and prevent their non-uniform re-distribution or coalescence during processing.

2. EXPERIMENTAL PROGRAMME

Two different materials were prepared with the aim of achieving high creep resistance and to make them suitable for mini-thixoforming. Compacts produced by various techniques were then processed in the semi-solid state under various conditions. Thanks to specially engineered heating equipment and a sophisticated control of the mini-thixoforming process, temperature and deformation schedules with steep changes in parameters can be implemented to high precision without overshooting the prescribed parameters.

The purpose of the development was to find suitable parameters for the entire technology chain to make it effective and to achieve compact microstructures with as few defects as possible and with fine particles uniformly distributed in the Fe-Al-based matrix.

2.1 Preparation of Powders

The initial particle size of the powders of both experimental materials (A and B) with a metal matrix containing Fe and Al and dispersed oxides was 30 μm. The powders were prepared by mechanical alloying with admission of air (**Fig. 1**). The powder prepared in this fashion was mechanically worked prior to compacting. The HPT method (High Pressure Torsion) was applied to the A material. It is one of the SPD (Severe Plastic Deformation) techniques and allows extremely high deformation to be applied to materials in the form of flat discs. The strain field within HPT-formed parts is very inhomogeneous. The variance in the compaction and strain was confirmed by microscopic observation. In the area of the highest-intensity deformation, which is near the disc edge, the compact contained numerous cracks and defects (**Fig. 2**). The hardness in these locations reached 730 HV10. From these discs cylinders of 6 mm diameter and 25 mm height were prepared using electrical discharge cutting (**Fig. 3**). Conical tips were electron-beam-welded on the cylinder faces in a vacuum to allow the specimens to be placed between electrodes for processing. These electrodes provided heating of these specimens.

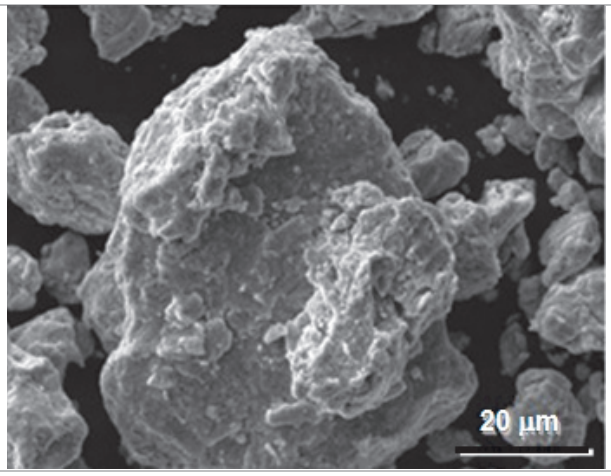

Fig. 1 Powder for the A material prepared by mechanical alloying

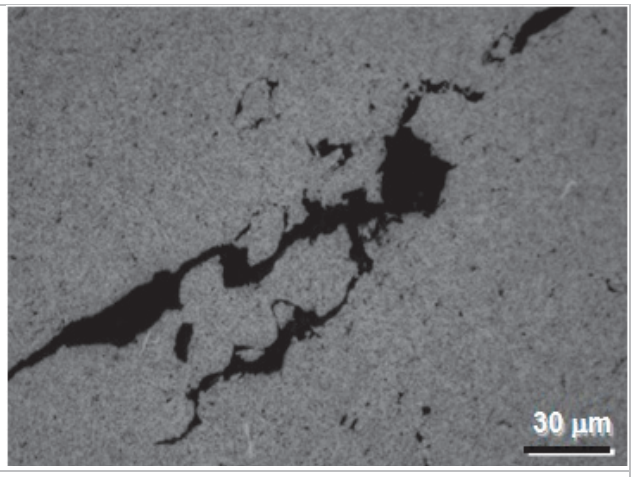

Fig. 2 Microstructure of the metal-oxide semi-product after HPT, material A

The B material was made from the same base metal but contained a different oxide. The powder was hot compacted in a press. In a vacuum chamber, the powder was pressed into a 32 mm-diameter steel container which was then sealed. After heating to 1150 °C in a furnace, the container was placed in a tool, in which it was compressed from its initial height of 50 mm to the final 12 mm and a diameter of 50 mm. The resulting

microstructure consisted of ferrite matrix and fine dispersed oxides and had a hardness of 461 HV10. The material contained inhomogeneities, most of which were located on contacts of compacted grains. Compacts obtained in this manner were used for making specimens for semi-solid processing, as with the A material.

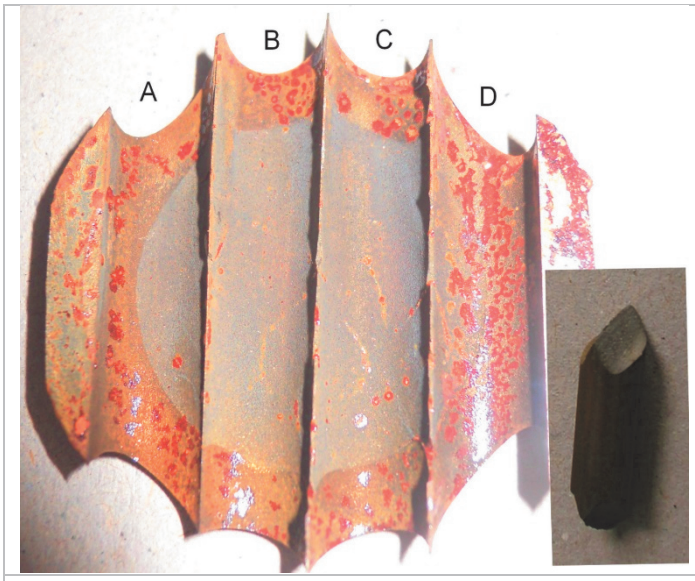

Fig. 3 Remnants of HPT-compacted discs after cut of the cylinders, material A

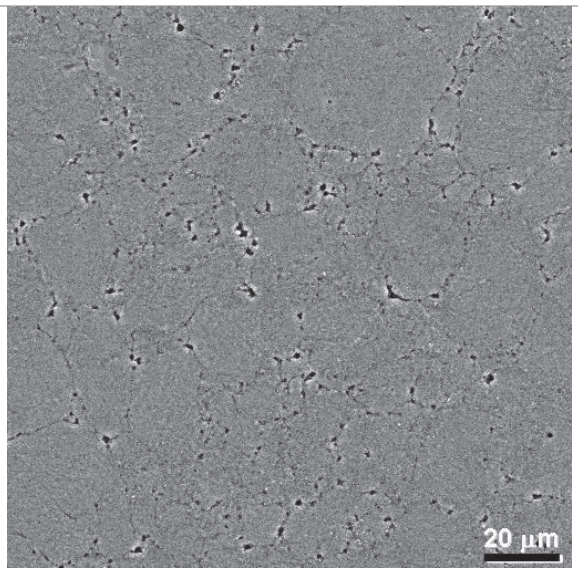

Fig. 4 Microstructure of the metal-oxide compact obtained by hot compacting in a press, material B

2.2 Semi-Solid Processing

Material A

As the specimens were welded together from three parts, where the central portion was from a different material than the ends, the heating parameters had to be found experimentally. The purpose was to achieve a uniform temperature field within the specimen during heating. First, the specimens were heated between electrodes without the die. In this arrangement, the temperature field can be measured using a thermal imaging camera and the temperature uniformity in the active part of the specimen can be assessed. The temperature of the active part of the specimen was gradually raised to 1400 °C. Identical parameters were then used for heating the specimen inside the die. The specimens were processed inside the cavity of a titanium die. The die is the standard mini-thixoforming tool used at the Research Centre of Forming Technology at the University of West Bohemia in Pilsen [5, 6]. The specimen temperature was measured by means of a rhodium-platinum-rhodium thermocouple welded on to the specimen surface. Based on previous experiments [3, 4], the forming temperature of 1400 °C was chosen. The schedule with the lowest heating rate comprised 68-second heating and a 10-second holding time. The material was then compressed using a force between 7 and 8 kN. It was left to solidify and cool down inside the die.

Material B

Specimens with the central part made from material B were processed in an analogous manner. First, heating to 1450 °C and deformation of the specimen in the titanium die were tried. The heating took 70 seconds and was followed by holding for 5 seconds. The heating plot consisted of two linear segments. Up to 1215 °C, the heating rate was 27 K/s. Above this temperature, the heating rate was reduced to obtain as homogeneous as possible temperature field throughout the specimen. The specimen was deformed using a force of 7 kN with a stroke of 7 mm. Additional tests were conducted without the die and the process could be observed directly. It was found that the uniformity of the temperature field and the effectiveness of the heating process depended

on the quality of the welds. Where the weld contained a defect and the resulting electrical conductivity of the part was poor, the prescribed heating temperature was not reached. Specimens which reached the prescribed temperature of 1450 °C were compressed by 5 mm using a force of 0.5 kN.

3. RESULTS AND DISCUSSION

Material A

Where the anticipated process parameters were achieved and the semi-solid processing at 1400 °C was performed, a relatively uniform microstructure with relatively homogeneously dispersed oxides was obtained. The resulting hardness was 460 HV5 (**Fig. 5**). The material contained small defects related to the solidification process.

Where the heating temperature was only 1000 °C, the resulting microstructure consisted of the Fe-Al solid solution and a dispersion of very fine globular oxides (**Fig. 6**) but the particles created by mechanical alloying were not compacted adequately (**Fig. 6**). Consequently, the resulting material was very brittle. The hardness of the central part of the specimen was 454 HV 5.

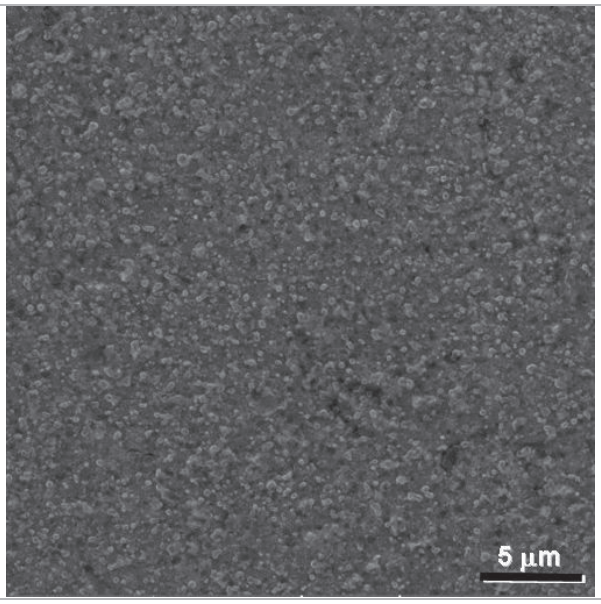

Fig. 5 Material A after semi-solid processing at 1400 °C

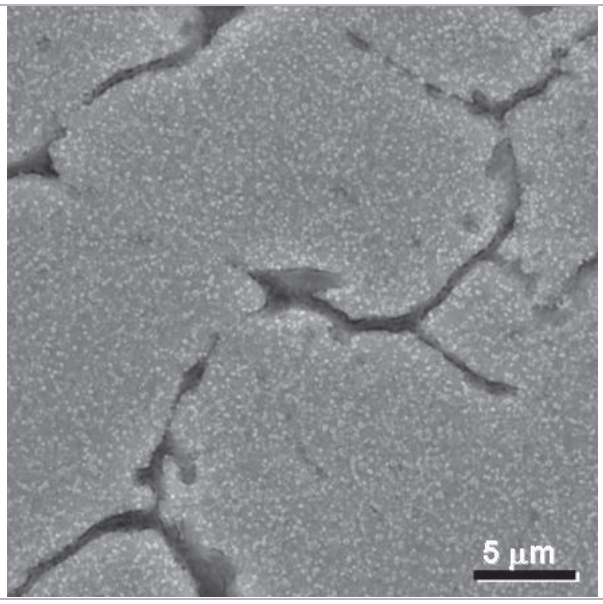

Fig. 6 Material A after processing at 1000 °C

Material B

The best results to date, i.e. uniform microstructures without major defects, were achieved by means of the schedule with the processing temperature of 1450 °C (**Fig. 7**). The high heating temperature had an adverse effect on the morphology of oxide particles, causing them to coalesce and coarsen. Consequently, shorter heating times will be used in future experiments. Hardness values were close to 363 HV10. With the heating temperature of only 1313 °C, ferrite matrix with a dispersion of fine oxide particles and a hardness of 447 HV10 was obtained (**Fig. 8**). However, the material was not fully compacted, as it did not undergo the short deformation in the semi-solid state.

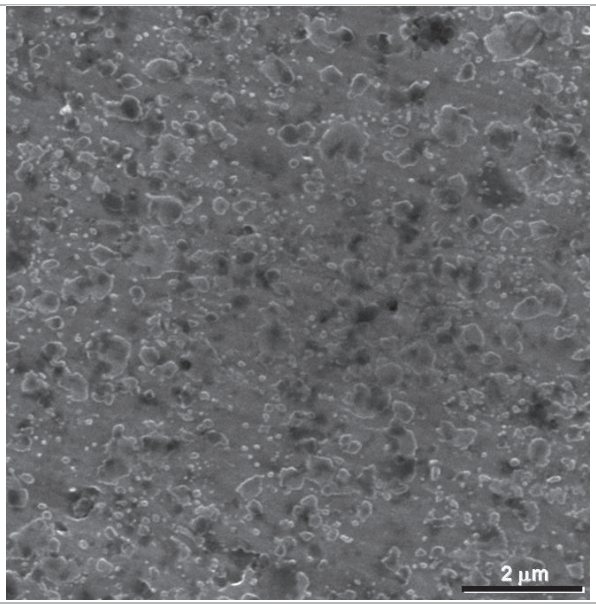

Fig. 7 Material B after semi-solid processing at 1450 °C

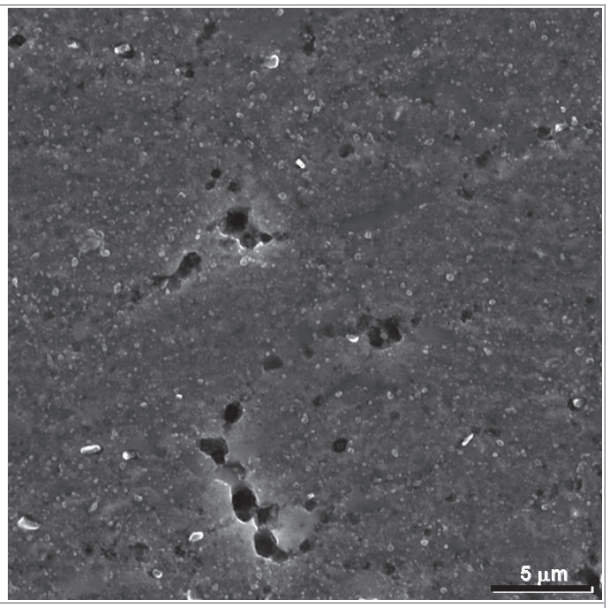

Fig. 8 Material B after processing at 1315 °C

CONCLUSION

The present paper outlines the potential of hot shaping of oxide dispersion-strengthened materials with high creep resistance. Two materials were processed using experimental schedules. The two materials differed from each other in the oxide used and in the compact preparation procedure. The advantage of both materials is the low cost of the input materials.

The experimental processing chain comprised mechanical alloying, compacting by means of various techniques involving plastic deformation and the final mini-thixoforming step. The purpose of the effort was to obtain a material with metal matrix strengthened with uniformly dispersed fine oxides. The matrix should substantially retain its uniformity during a short transition through semi-solid state and the oxides should not coalesce. Materials of this type have the required potential for making intricately shaped miniature parts by mini-thixoforming. In order to explore their applicability, two different compacting methods with plastic deformation were used in the processing chain. One was based on the HPT technique and the other involved severe warm compression.

All technological specifications were met in processing both materials. Compact microstructures with minimum numbers of defects and with acceptable distribution of oxide particles were obtained. On the whole, finer precipitates with more uniform distribution were obtained in the A material. This pilot project showed that this particular process chain offers a great potential. Follow-up research tasks will focus on shortening the heating and the semi-solid processing times to inhibit diffusion, as it promotes undesirable coalescence, coarsening and clustering of oxide particles.

ACKNOWLEDGEMENTS

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