Microstructure of CoAl-Co Intermetallic-Matrix Composite After Different Heat Treatments

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ABSTRACT

CoAl-Co intermetallic-matrix composite was synthesized from a dense homogeneous blend of micron size elemental Co and Al powders via solid state reactions. Consolidation of blends was performed by cold sintering–highpressure consolidation at ambient temperature. Subsequent reasonably short two-step heat treatment in the temperature range 600°C–800°C resulted in solid state reactive synthesis of the CoAl-Co composite. The synthesized material was subjected to different heat treatments. Annealing in the temperature range 900°C–1200°C does not fundamentally change the microstructure of the synthesized material but results in an increase of microhardness. The fracture is intergranular. Heat treatment at 1250°C followed by quenching and annealing in the temperature range 400°C–900°C results in a change of the composite microstructure and in a change of the fracture mode to a transgranular one. The microstructure and phase composition of the materials obtained were characterized employing X-ray diffraction and SEM/EDS.

INTRODUCTION

Aluminium-base intermetallics and the intermetallic matrix composites offer the potential of combining increased specific strength and reliability with an excellent oxidation resistance at high operating temperatures /1-7/. This is primarily due to the fact that, compared to conventional high temperature engine materials, such as Ni-, Co- and Fe-base superalloys, these alloys have a favorable combination of high melting point, low density and significant elevated-temperature strength. Much interest has been centered on the titanium aluminides and on the nickel aluminide Ni_3Al which possess a positive temperature dependence of strength and are used for the development of new lightweight materials.

Equiatomic aluminides of cobalt and nickel, CoAl and NiAl, possess a distinct advantage over Ni_3Al from the point of view of melting temperature, density, elastic modulus and potential for high temperature oxidation resistance and over titanium aluminides from the point of view of melting temperature and elastic modulus. One of the main drawbacks of these materials is room-temperature brittleness and low fracture toughness that have limited their use as strengthening phases in conventional alloys. The main approaches to overcoming low-temperature brittleness are microalloying, grain refinement, alloying to produce multiphase structure and the use of reinforcements by ductile phase. For titanium aluminides, promising results were obtained for dual phase structures /8-12/, where one of the phases is ductile.

The Co-Al binary system holds considerable promise for the development of CoAl-Co intermetallic matrix composite containing Co as a ductile phase, since there are no intermediate phases between CoAl and Co (see Fig. 1). Additionally, the considerably increased solubility of Co in CoAl at temperatures higher than 800°C may probably allow variance of the microstructure of CoAl-Co alloy by different heat treatments.

Conventional casting of this alloy is difficult due to its high melting temperature. In addition, cast intermetallics are usually not suitable for subsequent cold or hot working because of inhomogeneities in the solidification microstructure along with micro- and macrosegregation /12/, whereas powder metallurgy

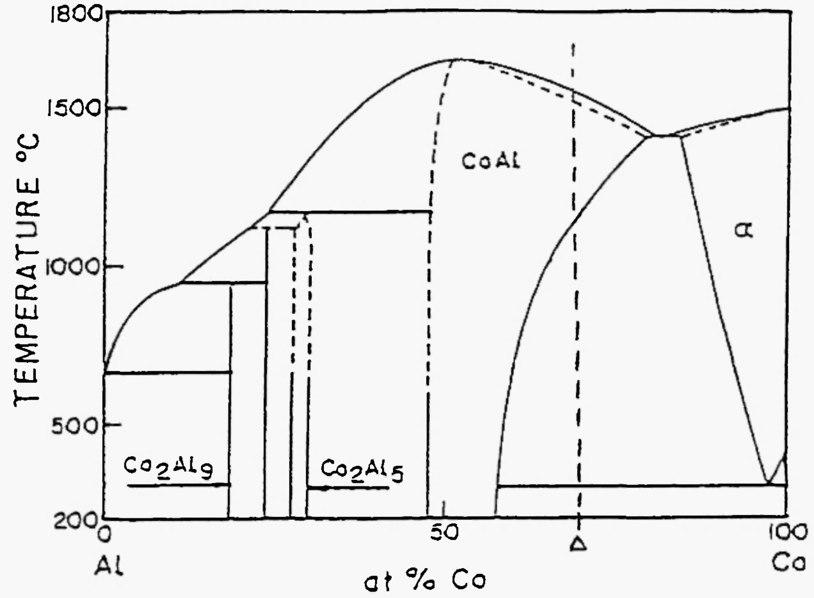

Fig. 1: Co-Al binary phase diagram.

allows fabrication of near net shape intermetallic products. In this work, the elemental powder approach was used to produce CoAl-Co intermetallic matrix composite. This approach was successfully used for synthesis of several intermetallics, including CoAl /13/. With micron/submicron elemental powders diffusion distances are short so that homogenization and/or synthesis of new phases can be completed over a much shorter time and, sometimes, at a lower temperature than with conventional size powders (> 40 μm). In the present work, the microstructure, phase composition and microhardness of the material obtained after synthesis and after different heat treatments were characterized.

EXPERIMENTAL

In the present research, ultrafine (< 1 μm) powders of Co from MHD and 5 μm size powders of Al from Cerac were used. Homogeneous powder blends of the composition corresponding to CoAl-Co two phase region on the equilibrium Co-Al phase diagram (84 wt.% of Co, 16 wt.% of Al) were prepared employing a high energy attritor (Union Process). A low ratio of grinding media to powder 2:1 was used to avoid mechanical alloying of SHS during milling.

Compacts of about 70% of theoretical density with an interconnected system of pores were treated at 400°C in hydrogen flow to remove the oxide layer from the surface of Co particles as well as of hydrites from the surface of Al particles /14/. After reduction treatment, compacts were cold-sintered/consolidated to full density in rigid dies at pressures up to 3 GPa (specimen size – 18 x 18 x 1.5 mm). Solid state synthesis of CoAl/Co composite was performed by two step heat treatment: 5 min at 600°C followed by 15 min at 800°C. In the first step, cold-sintered specimens were placed between heated (600°C) punches on an Instron testing machine (compression mode, stress applied s = 500 MPa). All the initial Al was consumed and several intermetallic phases were formed during this heat treatment. The microhardness of such partially synthesized material was about 9800 MPa. To accomplish the synthesis, the specimens were heat-treated at 800°C in a vacuum furnace.

After that two series of heat treatments, A and B, were performed. In the A-series, the specimens were annealed at several temperatures in the temperature range 500°C-1200°C for one hour and quenched to room temperature. In the B-series, the specimens were initially heat-treated at 1250°C for 1 hour, quenched to room temperature and annealed for 1 hour at different temperatures in the temperature range 400°C–900°C.

X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive analysis (EDS) were used for characterization of microstructure and phase composition of the material. Microhardness of the material after different heat treatments was measured and compared.

RESULTS AND DISCUSSION

XRD spectra of material after synthesis heat treatment is shown in Fig. 2. Peaks corresponding to CoAl and FCC-Co are observed in the spectra. According to the equilibrium phase diagram, not FCC but HCP Co is stable at temperatures lower than 300°C. But the FCC-HCP transformation is known to be very sluggish in Co. Al atoms, which can be dissolved in FCC-Co in a much larger amount than in HCP Co, can additionally slow down this transformation.

The microstructure of synthesized material is shown in Fig. 3a. The material consists mainly of Co (3white regions) and dense CoAl phase (gray regions). Dark gray regions are porosive CoAl phase. The porosity develops during the reactive synthesis of intermetallic phases because of a very big difference in mobility of the Al and Co atoms participating in the formation of intermediate and resulting intermetallics. The microhardness of synthesized material was about 500 MPa.

For the A-series, microhardness of CoAl/Co composite after annealing at different temperatures for 1 hour is plotted in Fig. 4. The measured value of microhardness decreases in the temperature range 400°C–900°C and increases in the temperature range 900°C–1300°C, having a minimum value of about 350 MPa at 900°C. The descending part of the microhardness curve can suggest that some amount of intermediate Al-rich intermetallic phases, which very high microhardness, was retained in the material after two-step synthesis heat treatment. These phases, which are metastable for a given material composition, dissolve during further annealing. This results in a decrease in microhardness. The ascending part of the microhardness curve can be explained by solid solution hardening of CoAl intermetallic due to Co atoms. The equilibrium solubility of Co in CoAl grows considerably for temperatures higher than 800°C. Annealing of synthesized CoAl/Co composite at these temperatures for one hour results in dissolution of Co atoms in CoAl according to the equilibrium phase diagram. Quenching retains the high-temperature phase composition at room temperature, resulting in oversaturation of CoAl. The level of the oversaturation depends on annealing

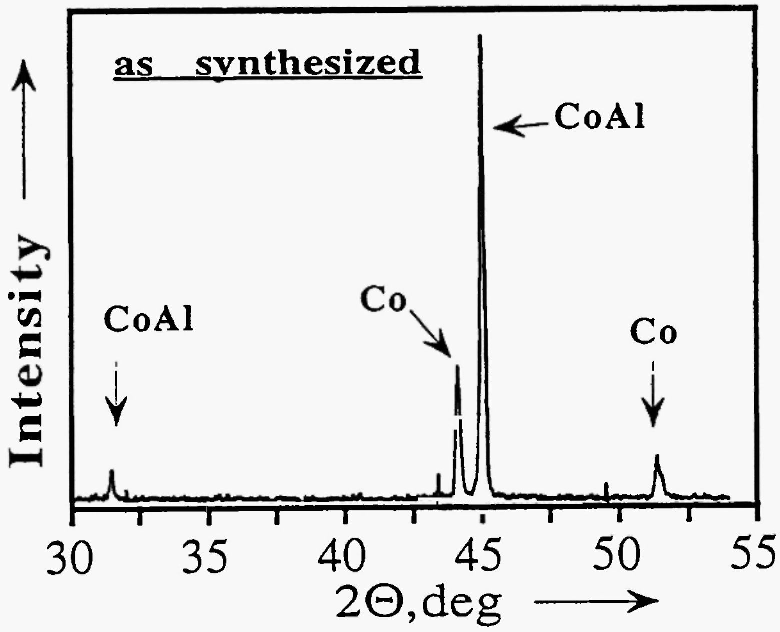

Fig. 2: X-ray spectra of CoAl-Co composite after synthesis.

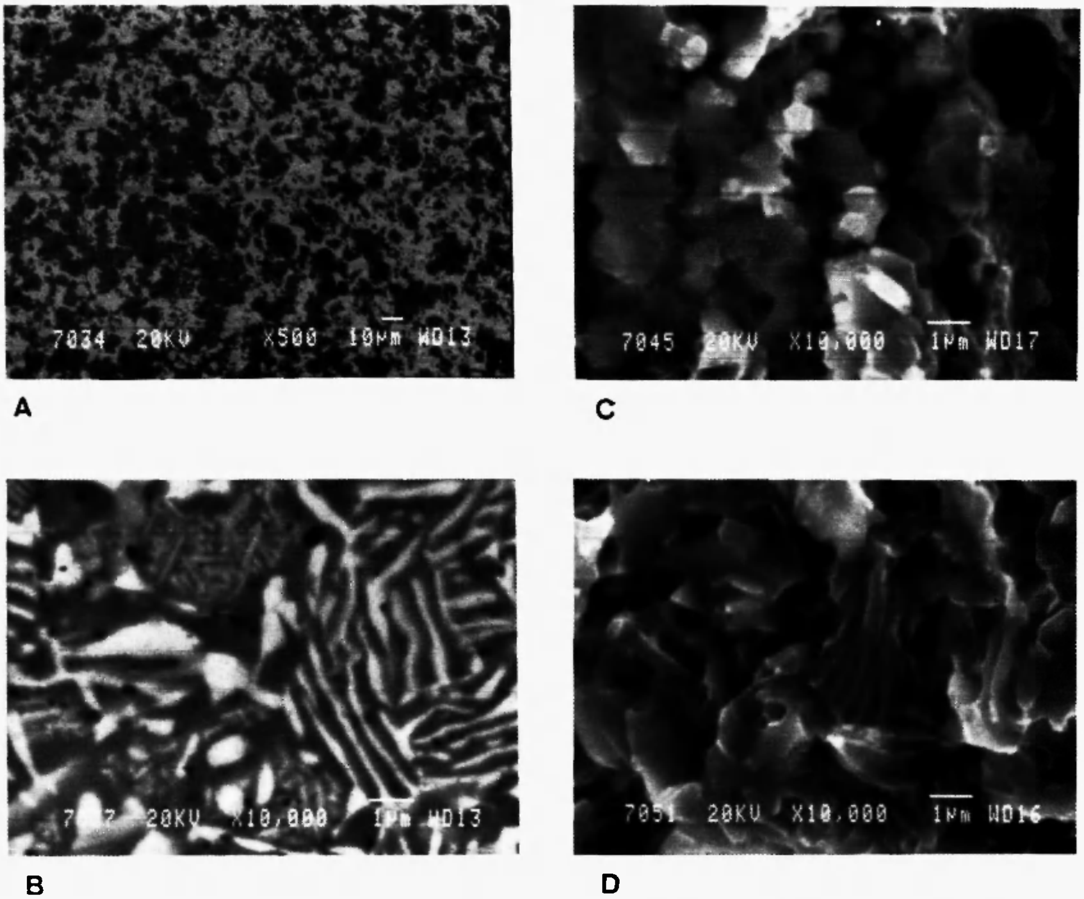

Fig. 3: Microstructure of CoAl-Co composite: a, c – as synthesized; b, d – after heat treatment: 1250°C x 1 h + quenching + 900°C x 1 h; (a, b – polished surface, SEM, back scattered electron image; c, d – fracture surface, SEM, secondary electron image).

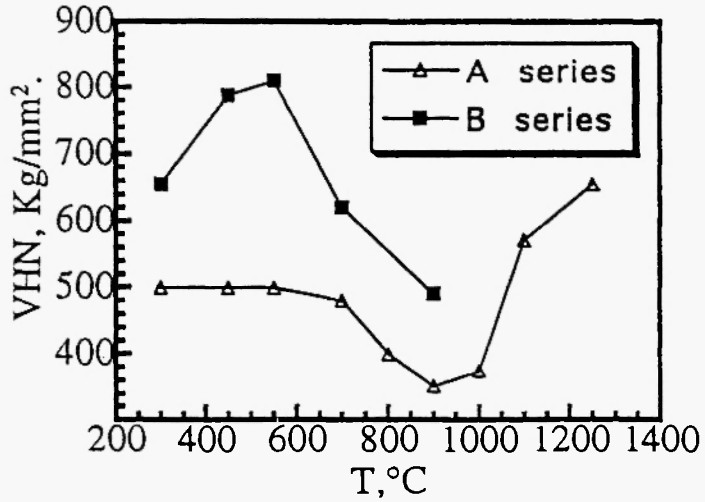

Fig. 4: Microhardness of CoAl-Co composite annealed at different temperatures: a – as synthesized; b – after additional heat treatment 1250°C x 1 h + quenching.

temperature and increases with the temperature, thus resulting in dependence of the room temperature microhardness of the composite on annealing temperature. According to SEM observation, the material has single phase CoAl composition after 1 hour annealing at 1300°C followed by quenching.

For the B-series, the microhardness of the material after quenching from 1250°C and annealing at different temperatures for 1 hour is plotted in Fig. 3. The measured value of microhardness increases in the temperature range 400°C–650°C and decreases in the temperature range 650°C–900°C, having a maximum value of about 810 MPa at 650°C. Increase in microhardness can be explained by precipitation hardening due to nucleation and growth of Co precipitates in the CoAl matrix. Overaging occurs at temperatures higher than 650°C. It should be noted that the microhardness of a material in series B (quenching from 1250°C + aging for 1 hour) is higher than that of a material in series A (annealing for 1 hour after synthesis) in the temperature range 400°C–900°C.

A typical microstructure of a specimen after quenching from 1250°C and aging at 900°C is shown in Fig. 3b. The material consists of a CoAl skeleton (gray regions) and dendrites of Co. The microstructure observed is fine, with dendrite branches highly elongated in one direction. It is known that the elastic constant and yield strength of Co is much smaller than that of CoAl. Additionally, specific volume grows during the transformation of supersaturated CoAl in CoAl-Co mixture. As a result, the shape of growing Co precipitates should be determined by the accommodation of elastic energy. It can be proposed that the branches of Co dendrites are placed along the "soft" crystallographic direction of CoAl.

Fractured surfaces of a specimen after synthesis prior to heat treatment and after quenching followed by aging at 900°C are shown in Fig. 3c and 3d, respectively. For synthesized material, the fracture is intergranular, Fig. 3c. It was previously shown by TEM /12/ that the grain size in synthesized CoAl is about 0.5–3 microns, i.e., comparable to the size of the starting elemental powders. The micrograph of the fractured surface of synthesized CoAl/Co composites supports the TEM observation. The microstructure observed is fine and the regions of fractured interface have a size comparable to that of the starting elemental powders. Unlike this material, transgranular fractures were observed in material quenched from 1250°C or aged, Fig. 3d. On these micrographs, the region can be seen where the fracture plane intersects the Co dendrite.

According to SEM observation of a quenched specimen, the grain size in the specimen after annealing at 1300°C and quenching is 3–6 microns. Additionally, it can be proposed that in aged specimens, each dendrite colony in Fig. 2b occupies no more than one grain, since a random orientation relationship can be expected in neighboring grains resulting in the absence of correlation of orientation of dendrites in neighboring grains. Different orientation of the "soft" crystallographic direction in neighboring grains should prevent "straight line" growth of Co precipitates or even stop it. According to SEM micrographs, the dendrite colonies have the size range 3–6 microns, which fits well the grain size range observed in quenched material. This supports the assumption that recrystallization takes place during heat treatment at 1250°C. It can be proposed that the cohesion is different for grain boundaries that were obtained as a result of contact of phases growing during the synthesis and for those produced during subsequent grain growth. Improved cohesion can be expected at recrystallized grain boundaries. This can result in change of fracture mode from intergranular for synthesized material to transgranular for heat-treated material.

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