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Microstructural development of powder metallurgy cobalt-based superalloys processed by field assisted sintering techniques (FAST)

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ABSTRACT

This study reports the microstructural evolution, physical and mechanical properties of cobalt-based superalloys processed from mechanical alloyed powders and consolidated by field-assisted sintering techniques (FAST). After an initial thermodynamic simulation of the ternary diagram by Thermocalc® to determine the composition, a sequential milling process was carried out at room temperature up to 40 h milling of two different alloying systems: Co-12Al-10W (at%) and Co-12Al-10W-2Ti-2Ta (at%). Characterization of the powders was performed by using X-Ray diffraction, scanning electron microscope with energy-dispersive X-ray spectroscopy (EDS) and particle-size analyzer. Consolidated samples were also characterized in terms of density, microhardness and hardness. In order to promote the dual γ/γ' microstructure, both alloys were aged after solution annealing heat treatment, improving a new route of consolidation with a new level of performance.

1. Introduction

The need to improve efficiency in the civil aviation industry entails development of modern gas turbines with a new generation of superalloys [1,2]. The discovery of the stable ternary $Co_3(Al,W)$ intermetallic compound with an ordered $L1_2$ precipitates structures by Sato et al. [3], provides a possible potential cobalt-based dual phase γ/γ' microstructures for superalloy performance at high temperatures. The dual phase microstructure consists of rafted γ'-cuboidal precipitates embedded in a continuous γ-matrix. It has been widely reported that cobalt-based alloys exhibit better behavior of hot corrosion, oxidation and wear resistance than Ni-base alloys [4–9]. Such alloys, however, were not used in the past because the high temperature strength is lower than Ni base alloys. Other discoveries have been reported for the cobalt-based system, such as Co_3Ti with a $L1_2$ structure [10,11] and Co_3Ta (γ-structure) [12,13]. None of these systems was capable of undergoing heat treatment at high temperatures. Currently, much research has been published that focuses on the analysis of the stability of the γ'-phase [14–17], the effect of distinct alloying elements in the stabilization of the γ or γ'-phase [18–21], and the partitioning effect [22–24], and how alloying elements may affect properties at high temperature [19,25,26]. With addition of -Ti and -Ta elements to the ternary system, a dual phase γ/γ' microstructure has been developed. Adding about 2 (at%) -Ti, raises the γ'-solvus temperature by > 90 °C and phase stability and volume fraction by 20% (similar effects are observed with the addition of 2 (at%) -Ta [22]). Both alloying elements are strong γ'-phase stabilizers [17,18], with the addition of -Ta significantly improving the high-temperature strength.

The use of CALPHAD (CALculation of PHAse Diagrams) allows new alloys to be prepared by using preliminary databases from other published researches. Although there is only a limited thermodynamic description of the phase stability of the γ' phase in the case of cobalt-based superalloys, it is possible to predict the ternary compound of Co-Al-W with the data bases provided by Cui et al. [27], Yang et at [28], and Zhu et at [29]. It should be noted that in the three studies the metastable γ'-phase was described as a stable one. Current research shows that it is necessary to reassess the thermodynamic description of the scheme due to the metastable condition of the γ'-phase in the Co-Al-W system [30]. Building on other works, this paper focuses on the powder metallurgy (PM) route to fabricate a novel cobalt-based alloy. This is presented as a new and alternative route, giving a finer and smaller grain size with higher efficiency parameters than conventional casting routes.

2. Experimental procedure

Thermodynamic modeling (ThermoCalc 5®) was used to design the desired cobalt-based superalloy by using the data base offered by Cui et al. [27]. The elemental powders Co (Co6160) and W (W4105) were provided by Eurotungstene (France) and the Al powder by

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Table 1
Nominal composition in (at%) of Cobalt-based superalloys examined.

Label	Co	Al	W	Ta	Ti
CoAlW (Ternary)	Bal	12	10	–	–
Co-2Ti/Ta (Quinary)	Bal	12	10	2	2

SultzerMetzco (AL54NS) (Switzerland). The alloying element as pure -Ta powder, 99.98% and commercially pure -Ti powder (CPTi grade 4) with a particle size below 75 μm were provided, respectively, by Alfa Aesar Karlsruhe (Germany) and (GfE Metal und Materialien GmbH, Germany).

Once the Co alloy was defined by thermodynamic calculations, with the aim of increasing the solvus temperature of γ', the effect of -Ti and -Ta as alloying elements was examined by preparing mixtures (see: Table 1) to be mechanically alloyed afterwards. The content of -Ta and -Ti were set by following research published by Suzuki in [31]. Mechanical alloying (MA) was performed in a planetary ball mill (Planetary Pulverisette 6, FRITSCH), using hard metal Co-WC vessel and balls at a speed of 300 rpm with a ball-to-powder weight ratio of 10:1. Before the high-energy milling, the powder mixtures were mechanically blended for 30 min. The milling process was performed under Ar to maintain an inert atmosphere during the milling step. The vessel was continuously refilled during the process.

Particle size distribution was characterized by a Mastersizer 2000 (Malvern, United Kingdom) to discriminate the particle size, considering the d_{50} parameter. X-Ray diffraction (XRD) was performed in a Philips Panalytical X'Pert Pro MRD system and intensity versus 2θ plots was acquired with the angular range of the region between 30° and 100°, with a step size of 0.02° and step time of 2.4 s per step. The crystallite size and microstrain data were obtained by the Scherrer method through using X'Pert Highscore software.

The morphology and microstructure characteristics of the milled powders were analyzed by using a SEM Philips XL-30 and FEI TENEO equipped with an EDS system. Differential thermal analysis (DTA) was performed on a Setsys Evolution TGA & DTA/DSC (Setaram), under argon atmosphere for both Co alloys from room temperature up to 1550 °C with a step rate of 5 °C min^{-1}, detecting the phase transformation and melting point.

FAHP samples were consolidated in a Gleeble 3800 equipment (Dynamic System Inc, USA), applying simultaneously pressure and temperature, a continuous alternative current of low frequency heats the material by Joule effect. Prior to consolidation, the equipment allows a dilatometry mode to optimize the final thermal cycle. The milled powder was set into a cylindrical graphite die of 10 mm diameter for the consolidation. The process was performed under vacuum ~ 10^{-5} Pa up to 1250 °C for 10 min with a heating and cooling rates of 100 °C min^{-1} and approx. 3 °C min^{-1} respectively. The graphite die was gripped at a load of 5 MPa inside the vacuum chamber. When the temperature achieved 800 °C, the pressure was increased up to 80 MPa (the heating and cooling rates were, respectively, 100 °C min^{-1} and approximately 3 °C min^{-1}). The temperatures were recorded with a thermocouple placed, respectively, in a punch and the center of the graphite die. The densities of the consolidated samples were measured by pycnometer of He (AccuPyc II, 1340).

Once the dual phase was promoted, because the γ' and γ phase are crystallographically coherent, lattice misfit (δ) between them is an important parameter to state the hardening effect achieved depending on the morphology and composition of the precipitates. Misfit was calculated after XRD of treated samples, using the equation $\delta = 2(a_{\gamma'} - a_{\gamma})/(a_{\gamma'} + a_{\gamma})$ [32]. Typically tends to be spherical in alloys with near-zero misfit and become with cuboidal shape as this magnitude increases. Morphology of precipitates was studied by SEM. The volume fraction of the γ/γ' phase was studied using SEM micrographs, which were analyzed utilizing ImageJ® software. All samples were chemically etched utilizing Carapella's solution to reveal the dual γ/γ' microstructure.

In order to characterize the mechanical properties of the FAHP samples at room temperatures, a Zwick Roell microhardness tester with a Vickers diamond tip and load of 200 g ($HV_{0,2}$) and 1 kg (HV 1) was used, the data was analyzed by the hardness testing software ZHu HD. Nanoidentation tests were carried out by using a MTS Nanoidenter XP with a maximum load of 360 mN. The hardness and elastic moduli were obtained by an indentation depth between 500 nm and 1250 nm. Both values were determined from the unloading part of the force-depth curves with a minimum of 30 repetitions on each sample, according to the Oliver-Pharr method [33].

3. Results and discussion

3.1. Thermodynamic calculations

Thermodynamic calculation provide a more precise view of the ternary phase diagram and design the optimal composition of the cobalt-based γ/γ' dual phase microstructure. A ternary diagram assessment of Co-Al-W is show in Fig. 1. The phase diagram was determined by Thermocalc software® at 800, 900 and 1000 °C. It was found that γ' was stable at 800 and 900 °C. The constituent phases in the Co rich portion at 900 °C were the Co solid solution γ-(fcc Co), the β-phase (AlCo), χ-phase (Co_3W), μ-phase (Co_7W_6), and the ternary compound γ'-Co_3(Al,W) phase $L1_2$. Considering these constituents, the selected ternary composition is marked as a black point in the 900 °C isothermal section, with Co (bal), 12Al (at%) and 10 W (at%). A higher content of aluminum was selected in order to compensate for possible losses during consolidation of future heat treatments.

3.2. Compositional and microstructural analysis of the powders

The prealloyed powders were obtained by mechanical alloying

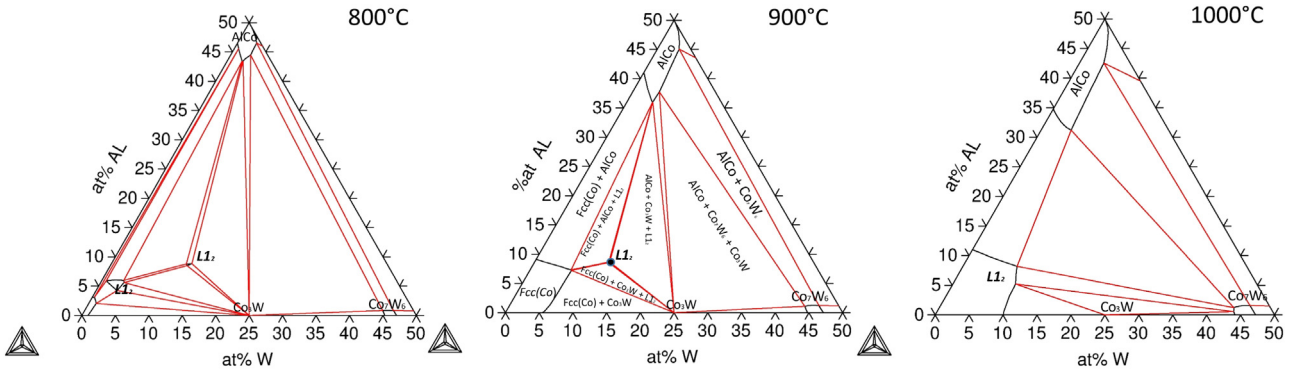

Fig. 1. Isothermal section of Co-Al-W ternary system at, respectively, 800, 900 and 1000 °C. The selected Co-Al-W composition is represented as a black point.