Synthesis of Haynes 25 Superalloy by Resistance Sintering Method

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Abstract

In the present study, Haynes 25cobalt-based superalloy produced by resistance sintering in open air under a uniaxial pressure of 250 MPa by using 4000A-3V for 7min. with Co, Cr, W, Ni, Fe and Mn powders was characterized. A heat treatment was made as follows: 1 h solution treatment at 980°C followed by air cooling and double aging heat treatment for 8 h at 720°C and then furnace cooling at 620°C and later ageing for 8 h. The microstructures and phase constituents were characterized by optical microscopy (OM), scanning electron microscopy (SEM+EDS) and X-ray diffraction (XRD). Hardness of sintered specimens was determined by using micro-hardness tester with a load of 100 gr for 10 s on polished cross-sectional surface. Microstructure examinations showed that a high density specimen with low porosity was achieved. The hardness of the samples was approximately 261±13 $HV_{0.1}$.Based on the Archimedes principle, the after sintering relative density of the samples was measured to be 94.3%.

Key words: Co-based superalloys, Electric current activated sintering

1. Introduction

Superalloys are heat-resistant alloys of nickel, nickel–iron or cobalt that exhibit a combination of mechanical strength and resistance to surface degradation generally unmatched by other metallic compounds [1]. Among these alloys, Haynes 25 (also known as alloy L-605) is a wrought, cobalt-based superalloy that exhibits excellent high-temperature strength, good ductility and good corrosion resistance [2]. At the same time superalloys a cobalt-nickel-chromium-tungsten alloy use for applications requiring high temperature strength, good oxidation and sulfidation resistance for prolonged times, ease of fabrication, weldability, an resistance to galling in service [3]. The strength and hardening behavior have been attributed largely to solid solution effects and carbides. Carbides are generally present even in an as-cooled material [2].It is widely used in established aircraft gas turbine engines for fabricated components, for balls and races in bearings, and for various industrial applications [4].

The Electric Current Activated Sintering (ECAS) is an ever growing class of versatile techniques for sintering particulate materials. ECAS is a consolidation method in which mechanical pressure is combined with electric and thermal fields to enhance inter-particle bonding and densification.This technique, loose powders or cold formed compact to be consolidated are inserted into a container which is heated to and then held at the desired temperature, while

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pressure is applied and maintained for a given period of time. Heat is provided by passing an electric current through the powders and/or their container, thus exploiting the consequent Joule effect. This technology is characterized by technological and economic advantages over conventional sintering methods such as faster heating rate, lower sintering temperature, shorter holding time, consolidation of difficult-to-sinter-powders, elimination of the need of sintering aids, no need of cold compaction, less sensitivity to initial powders characteristics, and marked comparative improvements in the properties of materials consolidated [5-8].

Haynes 25 superalloys have been produced through ingot metallurgy and some combination of hot/cold working and are readily weldable by most standard methods [9]. However, there is no available published work in literature about the superalloys of production by ECAS. The aim of this study is to investigate the production of Haynes 25 Co-based superalloy by one-step pressure assisted electric current activated sintering. The morphology and distribution of alloying elements within specimens were determined by scanning electron microscopy (SEM+EDS). In order to determine the phases and measure the micro-hardness of samples x-ray diffraction (XRD) method and micro-hardness tester with Vickers diamond indenter were utilized.

2. Experimental Procedures

The chemical composition of starting powders and their amounts in percentage of Haynes 25 superalloy produced in this study are given in Table 1 in weight %.

Table 1.The initial powder properties and chemical composition of Haynes 25 Co based superalloy

Powders	Properties of powders	Composition wt.%
Cobalt (Co)	< 44 μm, %99.9 purity	Balance
Chromium (Cr)	1-5 μm, %99.8 purity	20
Tungsten (W)	≤ 200 μm, >%99 purity	15
Nickel (Ni)	3-7 μm, %99.8 purity	10
Iron (Fe)	1-9 μm, %99.9 purity	1
Manganese (Mn)	< 10 μm, %99.6 purity	1.5

These powders were mixed according to chemical composition of the alloy. The mixed powders were placed in mould. Boron nitride (BN) powder was pasted on the die surfaces and punched to facilitate the separation of the specimen after sintering. The superalloy was produced by electric current activated sintering. The specimen was kept in open atmosphere prior to compression to closest the contact of powders and then the sample was compressed in a die with a compression load of 250 MPa for 1 min. A direct electric current (4000A-3V) was applied to sample with a pressure of 25 MPa for 7 min in order to produce superalloys. After sintering, the specimens were removed and cooled to room temperature in an open atmosphere. In order to balance the strength and ductility, the sample was exposed to the standard solution heat treatment at 980°C for 1h prior to quenching. Then two-steps ageing treatment consisting of 720°C for 8h with furnace cooling at 55°C h^{-1} to 620°C and holding at 620°C for 8h before air cooling to room temperature. The morphologies of the samples and the presence of the phases formed were examined by with

optical (OM) and scanning electron microscopy (SEM-EDS) and X-ray diffraction (XRD) analysis. The micro-hardness of the samples was measured by using a Vickers indentation technique with a load of 100 g on polished cross-sectional area of the superalloy. The relative density and porosity of the as-produced and heat treated samples were measured according to Archimedes' method.

3. Results and Discussion

Pressure-assisted electric current–activated sintering process has combined effects of both the electric current and pressure, so that, the desired can be obtained by simultaneously performing synthesis and consolidation in a single step. The electric current was used to supply the energy needed to complete the reaction and to generate the Haynes 25 superalloy in-situ. In order to decrease the amount of porosity and obtain a denser structure, the pressure is directly applied to the powders placed in the mould. The microstructures of synthesized and heat treated Haynes 25 superalloy samples were given in Fig. 1.(a,b). As shown in Fig.1(a,b), there are three distinct phases in the synthesized and heat treated samples which are white, grey and block shaped phases. In addition, the samples have smaller pores, but pores in the heat treated alloy are larger.

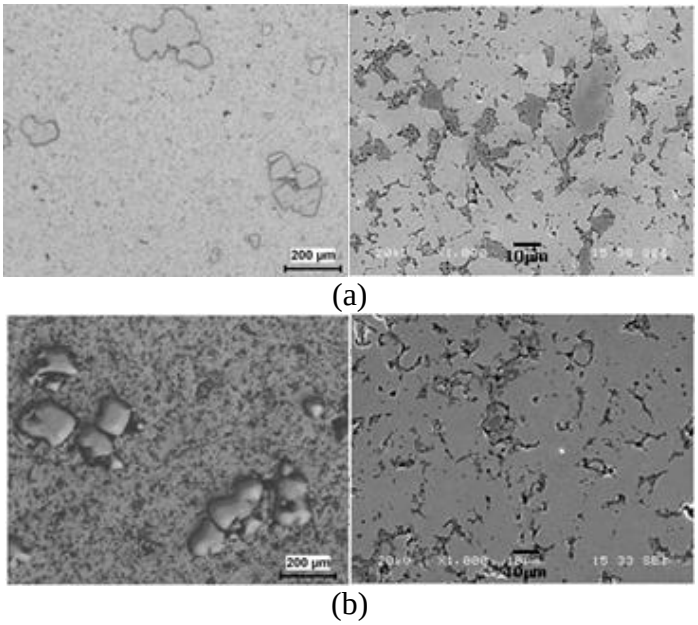


Figure 1. OM and SEM images of sintering samples (a) Synthesized (b) Heat treated

Three carbide phases are present in the Haynes 25 alloy. These are M_6C (fcc) $M_{23}C_6$ (fcc) and M_7C_3 (hexagonal), where M represents a metallic element such as tungsten. The Haynes 25 alloy also contains precipitates of Co_xW_y if it is aged at elevated temperatures. There are four types of Co_xW_y phases: Co_2W phase exhibits a hexagonal C_{14} type Laves structure (Pearson symbol; hP12) with the probable formula as $(Co,Ni)_2(Cr,W)$, α-Co_3W phase; $L1_2$-ordered fcc (Pearson symbol cP4), β-Co_3W phase; $D0_{19}$-ordered hexagonal (Pearson symbol hP8), γ-Co_7W_6; $D8_5$-hexagonal rhombohedral phase (Pearson symbol hR13). The different phases develop at specific aging temperatures over particular times: The Co_3W α- and β- phases were observed after aging

200 hours at and below 800 °C, where the Co_2W Laves phase and the $Co_7W_6\gamma$- phase are observed. The Co_2W Laves phase appeared after aging at 1050°C for only 10 hours. It was determined that the carbides, $M_{23}C_6$ and M_6C, developed very quickly with aging time of less than 2 hours. They were observed the earliest appearance of carbides at about 1050°C [3]. Fig. 2 shows the X-ray diffraction patterns of Haynes 25 alloy with double aging techniques. In this study, the XRD analyses of the heat treated Haynes 25 superalloy showed W, Co-Fe spinel, Co_3W and cobalt alloy matrix phases. Coarse W starting powder and short sintering time (7min.) was not sufficient to complete the reaction and thus prevented diffusion.

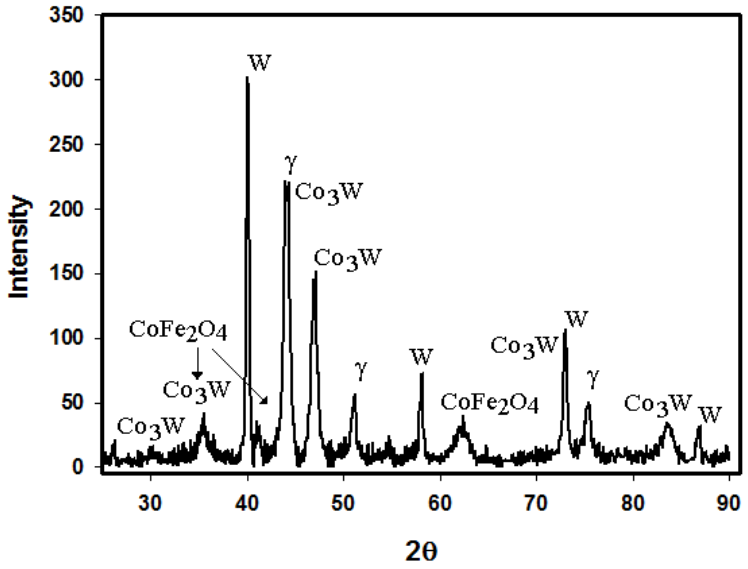
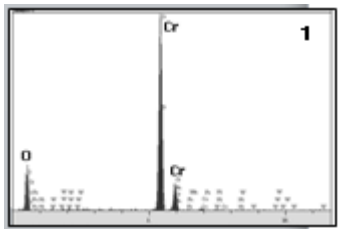


Figure 2. X-ray diffraction patterns of heat treated Haynes 25 sample.

The distribution of alloying elements in the synthesized and heat treated samples were determined by EDS analysis as shown in Figure 3 (a,b). According to EDS analyses of the synthesized and heat treated samples, it was observed that grey and white phases had containing elements given in Table 2.



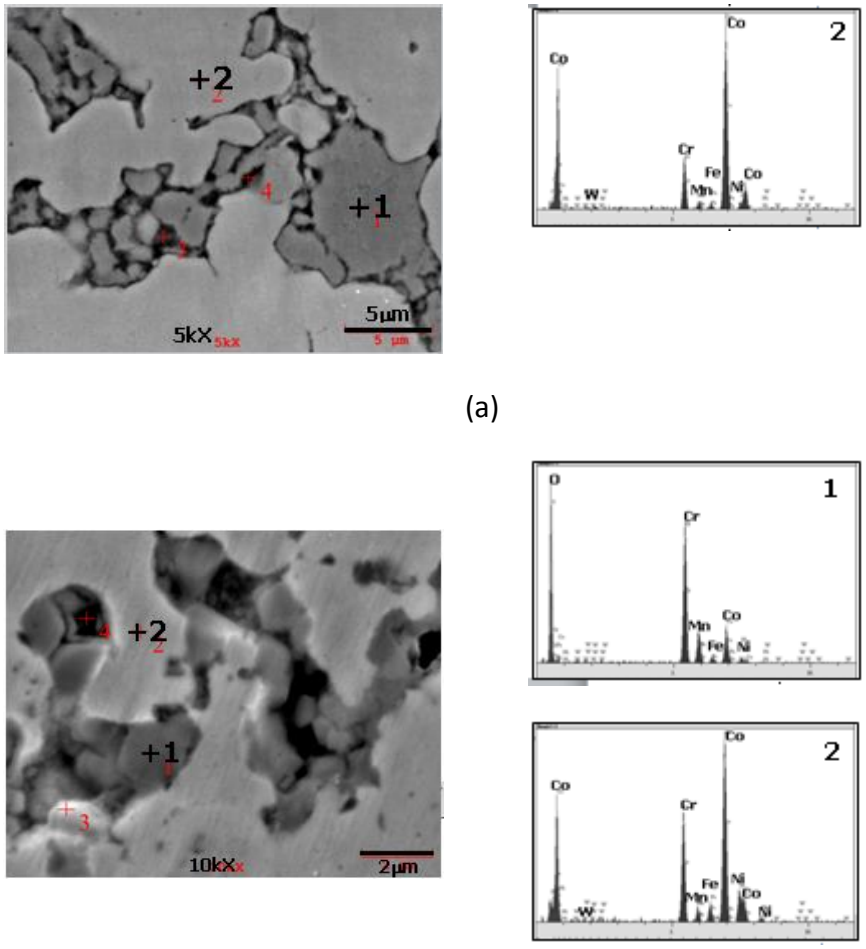


Figure 3.EDS analysis of Haynes 25 superalloy samples (a)synthesized (b) Heat treated

Table 2. The content of grey and white phases

Content of grey region (wt.%)							
	Cr	O	W	Co	Mn	Ni	Fe
Synthesized	78.8	14.6	3.8	1.7	-	-	-
Heat treated	39.6	32.9	1.6	16.9	5.1	2.2	1.9
Content of White region (wt.%)							
	Cr	O	W	Co	Mn	Ni	Fe
Synthesized	11.4	1.5	1.1	81.7	-	2.3	2.04
Heat treated	18.4	3.9	1	59.7	-	11.9	5

As seen in Table 2, the grey region is Cr-rich phase and white region is Co-rich phases. In grey phase region of heat treated sample, the amount of Co is higher compared to Cr, while Cr decreases synthesized sample. Also, Mn, Ni and Fe were detected in heat treated sample. In white phase region of heat treated sample there are large amount Cr, Niand Fe, on the other hand these

elements are less in synthesized sample. Block-shaped secondary phases were observed in the samples (Fig.1 (a,b)). A tungsten-rich phase was determined by EDS analysis (Fig. 5). It was seen that cobalt diffused towards this W-rich phase but sintering time was not sufficient due to coarse W powders.

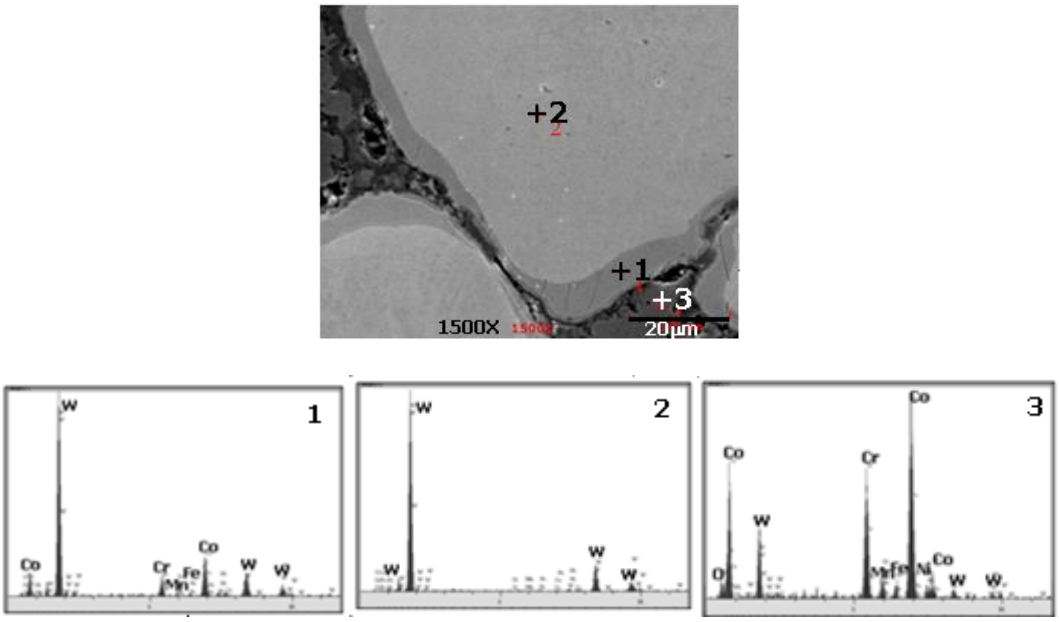


Figure5.EDS analysis of block-shaped phase in the synthesized and heat treated Haynes 25 superalloys.

The hardness of synthesized and heat treated Haynes 25 samples were measured using Vickers indentation with a load of 100 g. The results were approximately 212±5 $HV_{0.1}$ for synthesized and 261±13 $HV_{0.1}$, for heat treated samples. According to the literature, hardness values of the Haynes 25 superalloys in the range of 265-270 HV after aging for 5h at 595 °C [4]. The relative densities of the synthesized and heat treated Haynes 25 samples were calculated according to Archimedes' principle and the result was approximately 96.7 and 94.3%, respectively.

4. Conclusions

The following results can be derived from present study:

(a) Haynes 25 Co-based superalloy synthesized by Resistance sintering has low porosity.
(b) The presence of W, Co-Fe spinel, Co_3W and cobalt alloy matrix phases was confirmed by XRD analyses.
(c) The relative density is 96.7.3% for the synthesized and, 94.3% for the heat treated samples.
(d) The microhardness of materials measured by Vickers indenter was increased from 212±5$HV_{0.1}$ to 261±13 $HV_{0.1}$ due to double aging heat treatment.

Acknowledgments

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