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High entropy alloys obtained by field assisted powder metallurgy route: SPS and microwave heating

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HIGHLIGHTS

- Innovative microwave synthesis of high entropy alloys starting from metal powder.
- Comparison between two different powder metallurgy technique.
- Numerical simulation to gain a deeper understanding of the reaction.

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ABSTRACT

The aim of this work was to investigate the field assisted powder metallurgy route for producing HEAs at equimolar composition, i.e. FeCoNiCrAl, starting from metal powders. Both mixed, mechanically activated and mechanically alloyed powders have been used. The powders obtained by mechanical alloying were synthesized only by SPS, whereas the remaining ones were sintered by SPS or microwave heating. The investigated field assisted sintering techniques allowed an extremely short alloying time, high energy density on the load and negligible contamination by the surrounding environment. Both the conducted sintering-synthesis technology resulted not definitive to produce chemical homogeneity and to obtain a single stable structure. Thus a subsequently heat treatment was required. The post heat treatment, indeed, led to a single crystalline structure (FCC) and the material was fully recrystallized. After heat treatment samples are isomorphic: they exhibit two different phases with the same FCC cell, but different chemical composition, in detail Fe-Cr richer and Al-Ni richer. SPS-ed samples present a reduced porosity, while microwave processed ones are much more porous and this is reflected in the mechanical properties.

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1. Introduction

Since the introduction of the concept of high entropy alloys in 2004 [1,2] they have been attracting much attention across the world, due to their unique properties [3]. This new class of alloys, called also multi component alloys [4], where each one of the major elements has a concentration between 5 and 35 at% with high mixing entropy in their liquid state [5,6], can be tailored to enhance many promising properties, such as high hardness [7], wear resistance [8] excellent high and low-temperature strength [9–11] and in general good resistance to oxidation and corrosion [12].

According to the literature research [13–16,4] the most used processing routes for multi components alloy systems are:

- from the liquid state, e.g arc melting [17] and induction melting [18], where the obtained ingots are remelted under high vacuum at least three or four times to improve the homogeneity; more recently splat quenching [19], selective electron beam melting [20], laser [21,22] and direct laser (DLF) [13,16] technologies have been used as well
- from the solid state, using powder metallurgy, i.e. mechanical alloying [23] [24], spark plasma sintering [25]0
- from the gas state, such as sputtering techniques, i.e magnetron sputtering [26], plasma deposition [13].
- by electrochemical process [27] (used mainly for coatings).

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The alloys prepared from the liquid phase usually have many structural defects such as voids, porosity, and a generally low as-cast hardness [4]. Recently, further developments in the powder metallurgy route provided promising results [25,28] despite a higher pore density for samples produced by mechanical alloying (MA) followed by consolidation [29]. However, the solidification from the molten state is usually accompanied by micro-segregations, while by the MA process, more homogenous chemical distribution and solid solubility extension could be reached [15].

Hence, an ideal synthetic route to produce HEAs should guarantee short alloying time (achievable, for instance, applying high energy density on the load [28], leading to rapid melting [30] and reduced contamination by the surrounding environment), efficient cooling [31] and capability to operate in controlled atmosphere. Besides the aforementioned synthetic routes, such conditions can be achieved using high frequency electromagnetic fields, like in microwave heating [32], provided that the load is capable to couple with the incident electric and magnetic field. This technique belongs to the so called FAST - Field Assisted Sintering Techniques, like spark plasma sintering [33–35]. Scientific literature regarding the use of microwaves to prepare HEA is limited to few contributions [24,37,38], in which HEAs are prepared by microwave-assisted combustion synthesis, starting from oxidic precursors as raw materials (and hence having alumina as by product [34]) or by metallic powders mixtures [24,36–38]. Microwave assisted combustion synthesis of pure metal powders as reactants has already been used during last decade by some of the authors to prepare intermetallics [39], functionally graded materials [40], or to join dissimilar materials [41]. The advantage of applying microwaves to combustion synthesis reactions is the high purity of the products, the rapid ignition of the reaction [42], the possibility to control the products microstructure [43] and cooling rate after synthesis, especially in presence of ferromagnetic reactants [44]. When applied to HEA synthesis starting from metallic powders, such processing route allows the formation of a minimal fraction of liquid phase, thus leading to the possibility of achieving microwave assisted near-net-shape processing [24], able to overcome the limits of current melting technologies (defects formation) or conventional solid state ones (time demanding), but with the drawback of some residual porosity ascribable to the pressureless conditions used [37,38].

The aim of this study is to investigate the field assisted synthesis of FeCoNiCrAl starting from metal powders mixture and single phase formation obtained by subsequently heat treatment.

Among the several family of HEAs the FeCoNiCrAl has been selected for field assisted processing due to its peculiar precursors composition: at least one ferromagnetic element (Fe, Co, Ni) is present and a further heat contribution is expected during synthesis as a consequence of the presence of at least one highly reactive element couple, like Al-Ni, Al-Fe, Al-Co. Moreover, the use of Al is expected to have the synthesis initiated below 700 °C [37,45] roughly corresponding to the melting point of aluminum.

2. Materials and methods

The following elemental powders (Table 1), supplied by Sigma Aldrich, were exploited as reactants to prepare equiatomic FeCoCrNiAl.

Table 1
Composition of the metal powders used (BCC = body centered cubic; FCC = face centered cubic; HCP = Hexagonal close-packed arrangement).

Element	Purity (%)	Particle Size (μm)	Cell
Fe	97.00	<44	BCC
Co	99.80	<2	HCP
Ni	99.70	<5	FCC
Cr	99.00	<44	BCC
Al	99.00	<75	FCC

Two different routes to prepare the powder mixture were exploited:

- mixing for 20 min to homogenize the powders;
- mechanical activation by high energy milling for 1
- mechanosynthesis (prealloying) by high energy milling for 35 h (an interval longer than 15 h is required to achieve the mechanical alloying [24,45].

Both mixing and the mechanical alloying were carried out using a Planetary Ball Mill PM 100 by Retsch GmbH, at 250 rpm in an argon atmosphere, with steel balls, BPR 10:1 and cycles of 20 min milling followed by 5 min as break time to avoid overheating of the mixture.

The powders obtained by mechanical alloying were synthesized only by SPS, whereas the remaining ones were sintered by SPS or microwave heating.

In microwave synthesis, uniaxial pressing was used at 400 MPa to form reactive disc-shaped specimens of 20 mm diameter and 10 g as weight. A single-mode applicator based on the rectangular waveguide geometry (WR340) was used. The central part of the applicator presents predominant electric field conditions, while the regions near the side walls correspond to predominant maximum magnetic field conditions, for loads of small dimensions and not introducing major perturbations. In order to avoid excessive oxidation, a constant Ar flux (20 NmL/min) was blown into the single-mode applicator during experiments. The applicator is powered by a magnetron generator (MKS-Alter, Reggio Emilia, Italy), with an output power level ranging from 100 to 800 W, connected to a three-port circulator and to a three-stub tuner (MKS-Alter). A shorting plunger installed on the other side of the rectangular applicator allows to controllably modify the electromagnetic field distribution along the cavity. Temperature during heating tests was monitored and recorded at 1-s intervals, for load positioned in the predominant electric field region, using an optical pyrometer (IKS-T14-09, Sitel control Srl, Milan, Italy). The thermal synthesis, occurring with a strong exothermal event, was stopped immediately after the ignition of the reaction, in order to avoid possible annealing effects due to extended exposure to microwaves or high temperature.

In SPS, the powders were sintered in a DR.SINTER® SPS1050 (Sumitomo Coal & Mining, now SPS Syntex, Inc.) apparatus with graphite punches and dies. All samples have cylindrical geometry with a height of 5 mm and a diameter of 20 mm. SPS was performed at a nominal maximum temperature of 1000 °C or 1250 °C (measured with a thermocouple inserted into a blind hole in the die wall), with a uniaxial pressure of 60 MPa. The heating rate was 100 °C/min up to 950 °C and 50 °C/min up to the set sintering temperature. The maximum temperature and pressure were held for 5 min, before allowing the furnace to cool to room temperature.

All samples, after the preliminary consolidation operations with microwaves and SPS technological processes, were annealed at 1200 °C for 50 h in a tubular furnace, into a reactor containing titanium-shavings as getters, to reduce oxidative effects. The heat treatment was performed to improve samples homogeneity, leading to the single-phase formation of FCC solid solution.

The crystal structure was characterized by X-ray diffractometer (X'Pert PRO - PANAlytical) with Cu-Ka radiation and the microstructure was observed using scanning electron microscopy (SEM/ESEM - Quanta200 – FEI and SEM/FEG Nova NanoSEM 450 - FEI), after metallurgical preparation and etching by FeCl3+HCl solution. Instrumented nano indentation (CSM Instruments) was used to

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