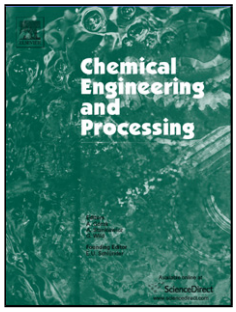

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MICROWAVE PROCESSING OF HIGH ENTROPY ALLOYS: A POWDER METALLURGY APPROACH

P. Veronesi[(1)], E. Colombini[(1)], R Rosa[(1)], C. Leonelli[(1)] and M. Garuti[(2)]

[(1)] Dept of Engineering "Enzo Ferrari", University of Modena and Reggio Emilia, Italy

[(2)] MKS, Alter Products, Via P. e M. Curie, 8, 42122 Reggio Emilia, Italy

Research Highlights:

- Innovative near net shape technology to synthesize high entropy alloys
- Excess Al favors microwave synthesis and homogenization in FeNiCoCrAl system
- Comparison between microwave heating, mechanical alloying and conventional furnace
- Best efficiency of microwave synthesis at ISM frequency of 2.45 GHz and 5.8 GHz
- Microwaves required the shortest time and lowest specific energy consumption

ABSTRACT

Microwaves at the ISM frequency of 2450 and 5800 MHz have been exploited to prepare FeCoNiCrAl-family high entropy alloys by direct heating of pressed mixtures of metal powders. The aim of this work is to explore a new microwave assisted near-net-shape technology, using powder metallurgy approach for the preparation of high entropy alloys, able to overcome the limits of current melting technologies (defects formation) or solid state ones (time demanding). Results show that direct microwave heating of the powder precursors occurs, and further heating generation is favored by the ignition of exothermal reactions in the compound. Microwave processing, exploited both for the ignition and sustaining of such reactions, has been compared to reactive sintering in laboratory furnace and mechanical alloying in a planetary ball milling. Results demonstrate that microwave required the shortest time and lowest energy consumption, thus it is promising time- and cost-saving synthetic route.

Keywords: microwave ignition, combustion synthesis, powder metallurgy

INTRODUCTION

High-entropy alloys (HEA) are a class of multi-component alloys composed of 5 or more principal constituent elements and each with a concentration between 5 and 35 atomic % [1].

These alloys have a tendency to form simple structures, like FCC and BCC, instead of intermetallic compounds [2]. Regardless of the HEA composition, this family of alloys shows several interesting features; in particular they tend to form simple solid solution phases with the possible presence of nanostructures or even amorphous structures [3], presenting Vickers hardness ranging from 100 to 1100 HV30, accompanied by a good thermal stability and excellent resistance to anneal softening [4]. Due to this broad spectrum of properties, the HEA have many potential applications [5], including mechanical parts and furnace parts requiring high strength, thermal stability and wear and oxidation resistance. Since the introduction of high entropy alloy concept in literature, several production techniques have been tried to synthesize these materials. According to the literature research, up to now, most of the production attempts followed one of the four following techniques: from the liquid state (arc melting, induction melting), from the solid state (mechanical alloying, powder metallurgy), from the gas state (sputtering techniques, mainly for coatings) and from electrochemical process (again mainly for coatings) [5]. The synthetic route should guarantee short alloying time (high energy density on the load, rapid melting and hence reduced contamination by the surrounding environment), efficient cooling and capability to operate in controlled atmosphere. Such conditions can be achieved using high frequency electromagnetic fields, like in microwave heating, provided the load is capable to couple with the incident electric and magnetic field. Scientific literature regarding the use of microwaves to prepare HEA is limited to only one contribution by Teng et al.[6] who synthesized FeCoNiCuAl HEA by microwave-assisted combustion synthesis, starting from oxidic precursors as raw materials (and hence having alumina as byproduct). 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 [7], functionally graded materials [8], or as a joining technique between dissimilar materials [9]. The advantage of applying microwaves to combustion synthesis reactions resulted in high purity of the products [10], rapid ignition of the reaction [11], possibility to control the products microstructure [12] and cooling rate after synthesis, especially in presence of ferromagnetic reactants [13].

MATERIALS AND METHODS

In this work non-equiatomic high entropy alloys have been prepared, starting from metallic powders mixtures, which include at least one ferromagnetic element (Fe, Co, Ni) and one highly reactive element couple, like Al-Ni, Al-Fe, Al-Co in order to improve heat generation due to both magnetic field contribution (microwave heating) and exothermal reaction contribution. Moreover,

the use of Al is expected to have the synthesis initiated below 700°C[14], roughly corresponding to the melting point of aluminum. Elemental powders have been used as reactants:

- Fe 97,0% purity, particle size less than 44 μm - Sigma-Aldrich
- Co 99.8% purity particle size less than 2 μm - Alfa Aesar
- Ni 99.7% purity particle size less than 5 μm - Sigma-Aldrich
- Cr 99.0% purity particle size less than 44 μm - Alfa Aesar
- Al 99.0% purity particle size less than 75 μm - Sigma-Aldrich

The proper amount of powders was weighed, to prepare a non-equiatomic mixture of:

1. $FeCoNiCrAl_{2.5}$
2. $FeCoNiCr_{0.5}Al_2$

and mixed in an agate jar for approximately 30 minutes. The excess Al, according to previous findings by the authors [14], allows to minimize segregation phenomena, due to the formation of an Al-rich liquid phase during heating, able to wet the remaining powders and promote reactions and diffusion. Uniaxial pressing was used at 300 MPa to form reactive disc-shaped specimens of 6 g as weight and 20 mm diameter and 5 mm height.

Two dominant factors establish the formation of a solid solution (SS), the enthalpy of mixing H_{mix} and atomic size mismatch δ according to Hume-Rothery rules. Therefore, Hume-Rothery rules are the earliest guide to the formation of SS alloys [15]. These are quantifiable by composition-weighted terms accounting for differences in atom radii (δ_r) and electronegativity (χ), as well as of the average Valence Electron Concentration (VEC). From a thermodynamic point of view, [16], the balance between the entropy and enthalpy contributions to the formation of a solid solution is captured by the parameter Ω, as defined in Eq.1

Eq.1 $\Omega = \frac{T_m \Delta S_{mix}}{\Delta H_{mix}}$

where ΔS_{mix} is the entropy of mixing and T_m the melting temperature. The most critical factor that decides whether an alloy crystallizes into BCC or FCC structure appears to be its VEC, as shown in Eq. 2 (the VEC of an alloy is calculated from the weighted average VEC of the constituent components) [17].

Eq. 2 $VEC = \sum_{i=1}^{N} x_i \, VEC_i$

According to previous studies on HEA [17][18][19], the characteristic parameters to achieve solid solutions are reported in Table 1[20] [21]. In this study, FeNiCoCrAl family of HEA have been selected, extending the Al content up to the limits of formation of the solid solutions (SS region in Fig. 1), and precisely the $FeCoNiCr_{0.5}Al_2$ and $FeCoNiCrAl_{2.5}$ HEAs, whose parameters are:

- for $FeCoNiCrAl_{2.5}$: δ =6.8 %, Ω=1.2, ΔH_{mix}=-16.1 [kJ/mol] [20], ΔS_{mix}=12.6 [J/molK], VEC=6.2, Tm=1510 [K], $\Delta \chi$=0.13 %.
- for $FeCoNiCr_{0.5}Al_2$: δ =6.7 %, Ω=1.2, ΔH_{mix}=-16.0 [kJ/mol] [20], ΔS_{mix}=12.7 [J/molK], VEC=6.6, Tm=1515 [K], $\Delta \chi$=0.13 %.

HEA synthesis experiments have been performed in different ways, in order to compare alternative synthetic routes:

- conventional heating in a muffle furnace, at 1100 °C, under a constant Ar flux (20 NmL/min), Optolab Mod. furnace, 1200 W power, for less than 2 minutes holding time at maximum temperature. 6 g disc shaped load
- microwave heating at 2450 MHz or 5800 MHz frequency, under a constant Ar flux (20 NmL/min), in TE10n single mode applicator, whose geometry has been described in details in previous work [13]. 6 g disc shaped load
- mechanical alloying in planetary milling with steel balls, using a Planetary Ball Mill PM 100 by Retsch GmbH, for times from 1 to 60 hours at 250 rpm in an argon atmosphere. A ball-to-powder mass ratio of 15:1 was used. Stearic acid was added as well, as the processing controlling agent (PCA) to avoid cold welding. 30g powder load

The choice of the single mode applicator lies in its possibility to expose the load to regions of predominant electric or magnetic field [22], even if both contributions have to be considered to heating, due to the perturbation of the electromagnetic field in the cavity provoked by the presence of the load [23].

Thermal synthesis, occurring with a strong exothermal event in case of excess Al content, 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. Mechanical alloying, instead, was conducted at different times, being impossible to externally detect events able to indicate the occurrence of the wanted reactions.
The crystal structure of mixed powders and as- synthesized alloys was characterized by X-ray diffractometer (X'Pert PRO - PANAlytical) with Cu-Ka radiation. The microstructure of the powders was observed using scanning electron microscopy (SEM, ESEM - Quanta200 – FEI), after cutting and polishing.

RESULTS AND DISCUSSION

The different synthetic routes investigated to synthesize the $FeCoNiCr_{0.5}Al_2$ HEA required different minimum average times and power to achieve a proper solid solution, as shown in Table 2, together with literature results referred to arc melting of similar alloys.

Mechanical alloying conducted for less than 15 hours did not allow to achieve the desired solid solution, as shown in Fig. 2, and hence a time of 60 hours was chosen for the comparison's sake. However, 15 hours will be used to assess the minimum specific energy consumption for the synthesis, as a conservative hypothesis that after such time the synthesis occurs.

The formation of the desired HEA in the system $FeCoNiCr_{0.5}Al_{2:}$ has been confirmed by X-ray diffraction, as shown in 3, where all the synthetic routes investigated are summarized. The peaks patterns are in agreement with literature results [24][25][26]. All patterns exhibit the solid solution formation, characterized by typical BCC structures, as predicted by VEC number <6.87 [17]. XRD analyses, supported also by EDS analyses, proved the absence of intermetallic phases, despite the excess aluminum used in some compositions.
Samples, after reactive sintering by microwave or conventional heating, retained their shape, indicating that the powder metallurgy approach is a suitable near-net shape technology. However, a pronounced porosity is left, as discussed later.

Based on these results, the microwave assisted synthesis of the $FeCoNiCrAl_{2.5}$ has been performed by microwave heating, to confirm the positive effects of the excess aluminum. In this case, microwave heating in predominant E or H field has been investigated as well.

Microwave assisted synthesis in predominant H field regions of the applicator required even shorter times, i.e.19 seconds, with no relevant difference in the final products, compared to predominant E-filed region. A higher magnification observation of the 45.5° peak shows that its Full Width at Half Maximum is slightly larger in case of E-field processing, possibly indicating that a less homogenous alloy, with strained lattice, has been achieved. This assumption has been confirmed by SEM-EDS observations, where regions with deviation from the theoretical stoichiometry have been observed. Such deviations regard mainly Fe and Cr, whose particle size is higher that the remaining powders (except for Al, which is not accounted for, since it is in the liquid phase during the synthesis). Fig. 5 shows the backscattered electron micrographs of the samples processed in predominant E- of H- field.

Microstructural investigations showed also that a pronounced porosity is achieved after synthesis, while retaining of the initial shape imparted during forming. This can be ascribed to the presence of the liquid phase (initially molten aluminum, then solutions with Al), which wets the solid particles, leaving porosity and generating also shrinkage porosity after solidification. However, not being predominant, the liquid phase does not deform the sample, which retains its shape. However, such porosity could be reduced by means of a modification of experimental set-up, allowing to impart a moderate pressure during synthesis, as already performed in previous works on aluminides synthesis [11][27].
The microstructure of $FeCoNiCr_{0.5}Al_2$ analyzed by SEM-EDS elemental maps is shown in Fig. 6. Microstructural analysis shows that all techniques lead to similar elemental distribution homogeneity.

According to previous work involving microwave assisted aluminides synthesis [28], it is possible to suppose that these microstructures are the result of the initial formation of molten Al, followed by exothermal reactions taking place between Fe-Al, Co-Al and Ni-Al, which provide a further heat contribution and promote the existence of the liquid phase.

The choice of operating at excess Al compared to equiatomic compositions proved successful in improving homogeneity of samples prepared by microwave heating: the presence of Al favors the liquid-solid reactions, highly exothermal, with consequent increase of the kinetic of reaction and synthesis temperature. Such exothermal contribution adds to the microwave heating one, which, alone would lead to much less homogenous structures, as shown in Fig. 7, referred to equiatomic FeCoNiCrAl, processed at 2.45 GHz at 300W.
The backscattered electrons SEM images of Fig. 7 still present regions with different grey scales, indicating a microstructure that is not perfectly homogenous, where parts of Fe and Cr powders are not reacted, as already demonstrated by authors in a previous work [21]. There is no evidence of differences between the microstructures of HEAs achieved by 2.45 GHz and 5,8 GHz, probably indicating that the reaction occurs in such a rapid and exothermal manner that it is dominated by the heat generated during synthesis, rather than on the input microwave power. The only relevant difference is in terms of reaction ignition time and duration, which is faster in case of 5.8 GHz. At this higher frequencies the power penetration depth in the pressed powders results halved with respect to the 2.45 GHz case, hence practically the same amount of power is dissipated into a much smaller volume. The result of this condition is that within the penetration depth the temperature is rapidly increased and hence the ignition conditions are reached earlier.

Given the similarity of results of the different techniques investigated, and using literature data, a tentative calculation of the specific energy consumption of each synthetic route was performed, and the results are compared and shown in Fig. 8. Among the thermal methods, microwaves are a fast and cost-saving technique, compared to traditional furnace (including the pre-heating time, which is time necessary to reach the temperature of ignition, at least 700 °C [14]) or arc melting However, it must be pointed out that in case of conventional furnace, the load is only partially filling the furnace, for approximatively 15% of the available volume. Hence, in case of larger sample, it is expected that the specific energy consumption could decrease by an order of magnitude. As a matter of fact, energy consumption is dominated by the heating of the furnace parts (heating elements, refractory lining), and only a minor fraction of the input power is used to heat he load. Nevertheless, under these conditions, microwave heating still

result the most energy saving approach, especially when using the 5.8 GHz frequency. However, the use of this frequency can pose problems when scaling up the process, due to the lack of high power generators operating at this frequency. The ball milling techniques resulted the longest and the more energy intensive; however, it has the advantage of producing HEAs in the powder form, and with small particle size, a condition which makes it suitable for further processing by conventional (or field-assisted) powder metallurgy techniques.

CONCLUSIONS

High entropy alloys belonging to the FeCoNiCrAl systems have been successfully prepared by microwave heating at 2450 and 5800 MHz of metallic powders compacts. In this study, high entropy alloys have been synthesized exploiting microwave heating and the supplementary heat contribution of exothermal reactions occurring during synthesis, in case of excess Al in the starting mixture. Comparison to other thermal (heating in furnace) and not thermal techniques (mechanical alloying) and to literature results (arc melting) allowed to evaluate the specific energy consumption of the different synthetic route. Microwave heating resulted the less energy intensive, in the experimental conditions investigated. The small dimensions of the microwave applicator used and the sort synthesis time makes this synthetic route interesting from a process intensification point of view, and the benefits become particularly relevant in case manufacturing of near net shape parts is required. The use of excess aluminum improved homogeneity of the final alloy, due to the presence of a liquid phase (initially made of molten aluminum) which improves the reaction kinetic. Samples characterization confirmed that the investigated thermal methods of powder metallurgy approach are suitable to retain the shape of the load imparted during forming by uniaxial pressing, despite the generation of an extended porosity. The minoritarian presence of the liquid phase and the extremely rapid and pressure-less microwave synthesis conditions did not lead to densification of the starting powders.

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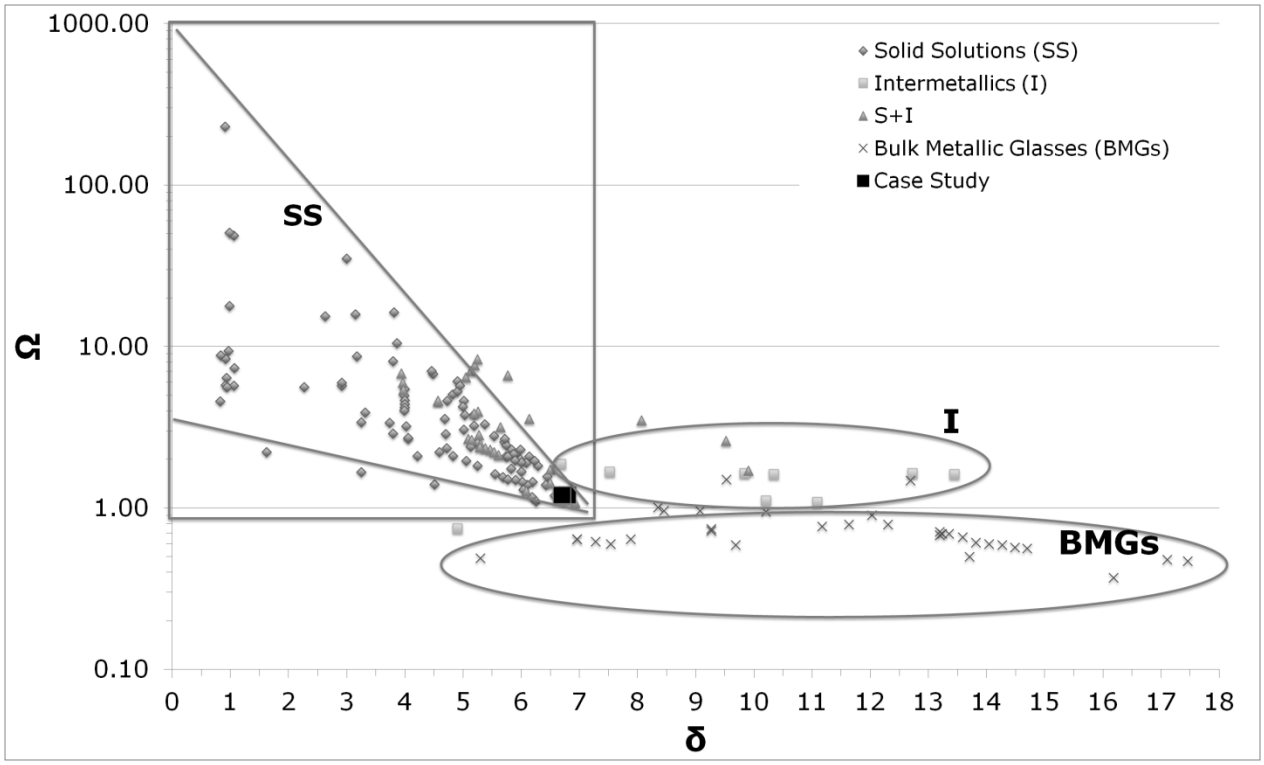


Fig. 1: Phase formation according to Ω and δ parameters and position of the HEAs subject of this work

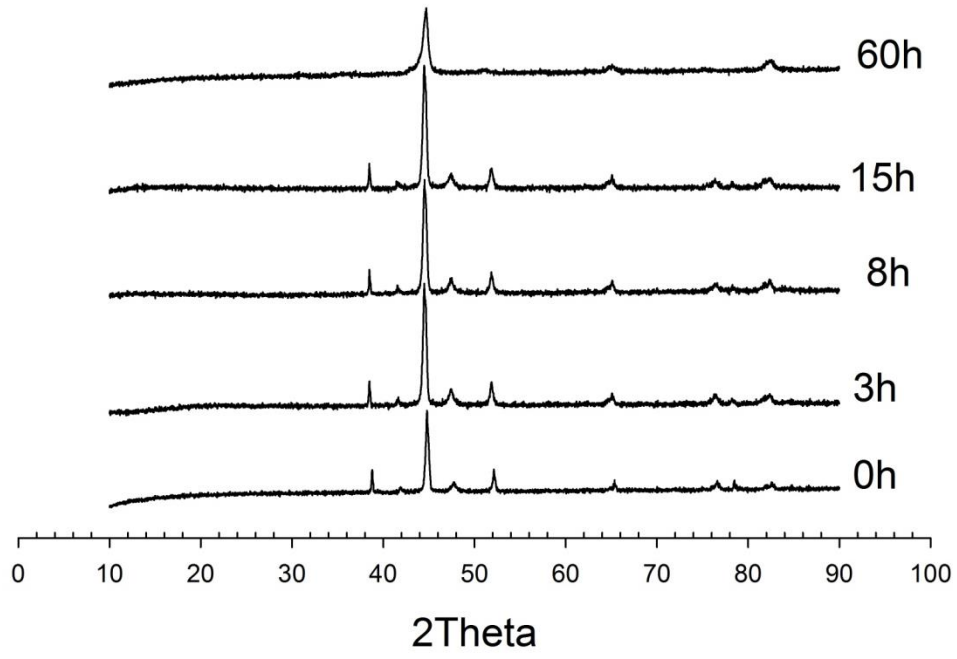


Fig. 2: XRD patterns at different time: 0-3-8-15-60 hours

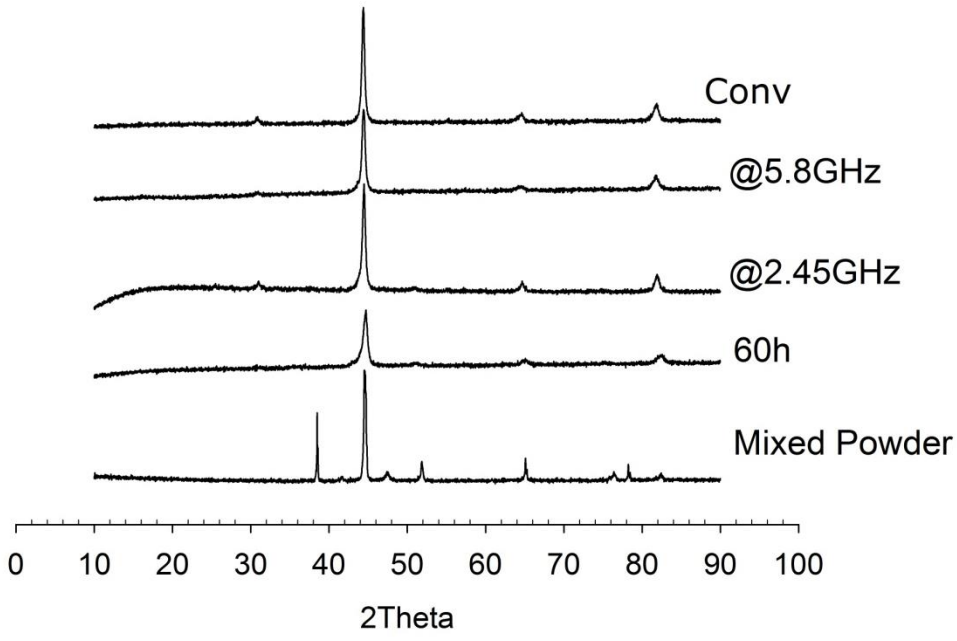


Fig. 3: XRD patterns of the $FeCoNiCr_{0.5}Al_2$ HEA, from top to down: conventional furnace, microwave heating at 5.8 GHz, microwave heating at 2.45 GHz, mechanical alloying after 60h milling time, starting powder mixture. The peak at 31° is ascribable to the mounting resin used

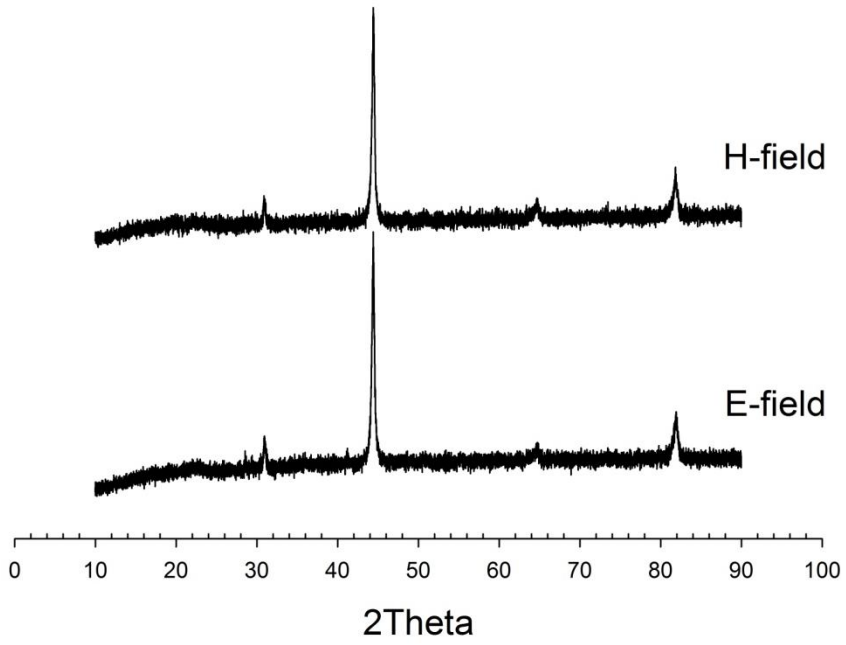


Fig. 4: XRD patterns of the $FeCoNiCrAl_{2.5}$ HEA, microwave synthesized in predominant E or H field, 2.45 GHz

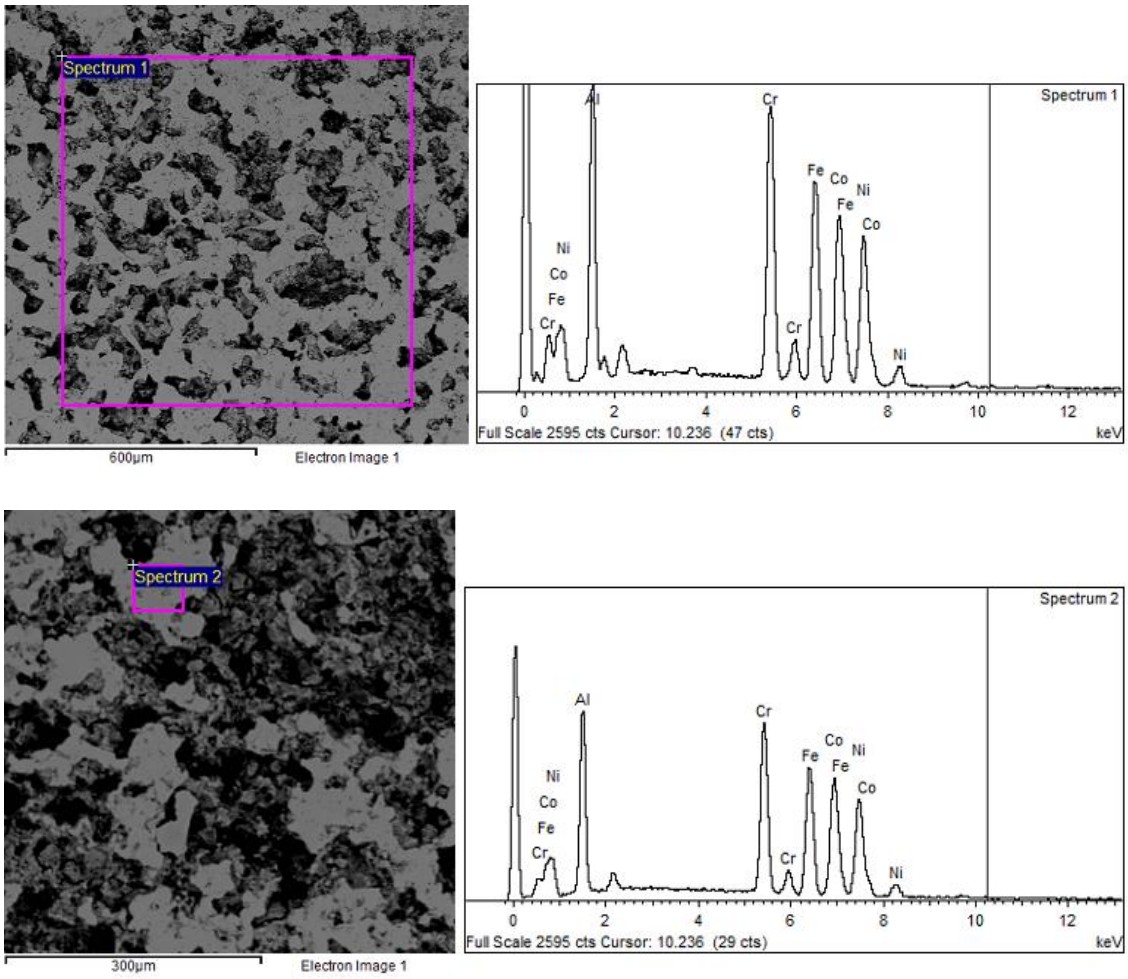


Fig. 5: BSE micrographs and EDS spectra of $FeCoNiCrAl_{2.5}$ HEA, microwave synthesized in predominant E (top) or H field (bottom), 2.45 GHz

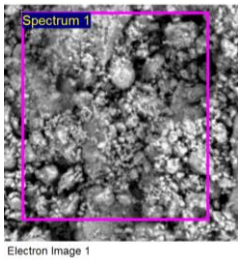
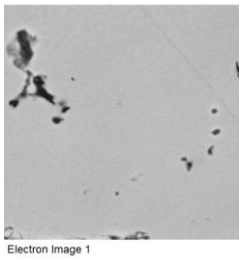
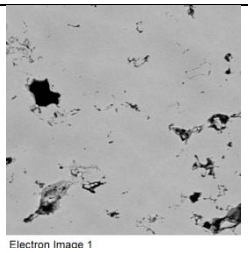
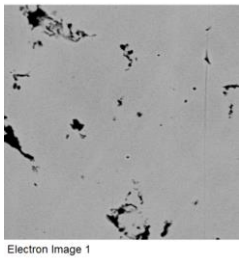
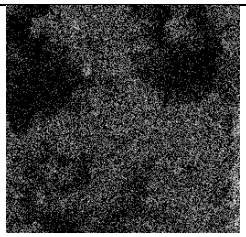
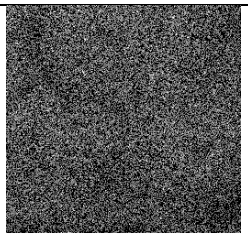
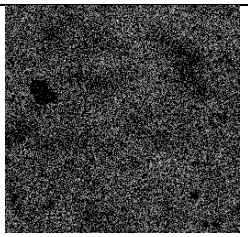
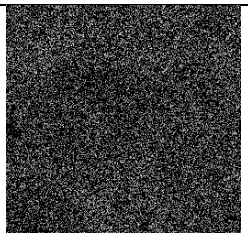
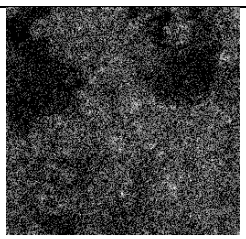
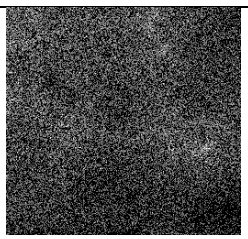
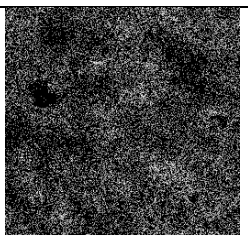
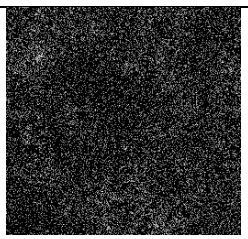
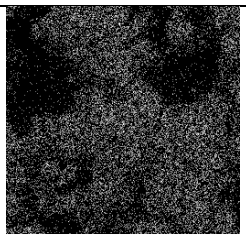
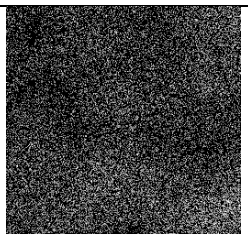
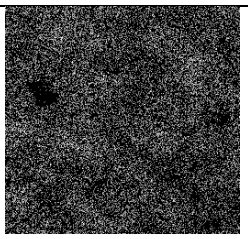
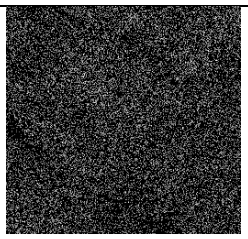
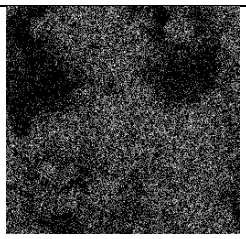
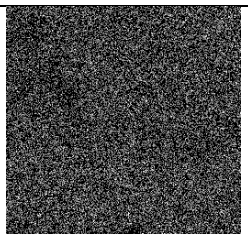
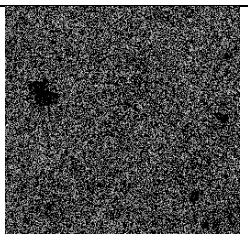
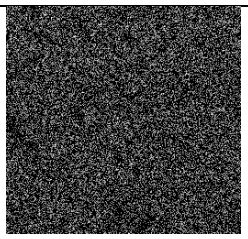
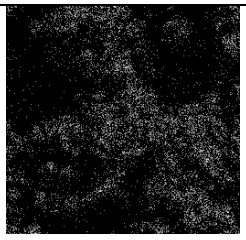
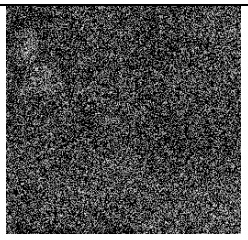
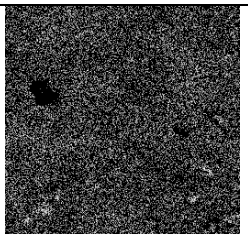
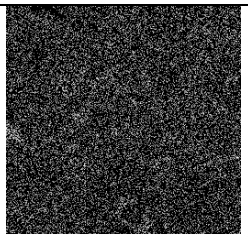
Elem.	Mechanical alloying	Conventional furnace	Microwaves, 2.45 GHz	Microwaves, 5.8 GHz
SEM				
Fe				
Cr				
Ni				
Co				
Al				

Fig. 6: SEM/EDS elemental maps of the $FeCoNiCr_{0.5}Al_2$ HEA synthesized with different routes

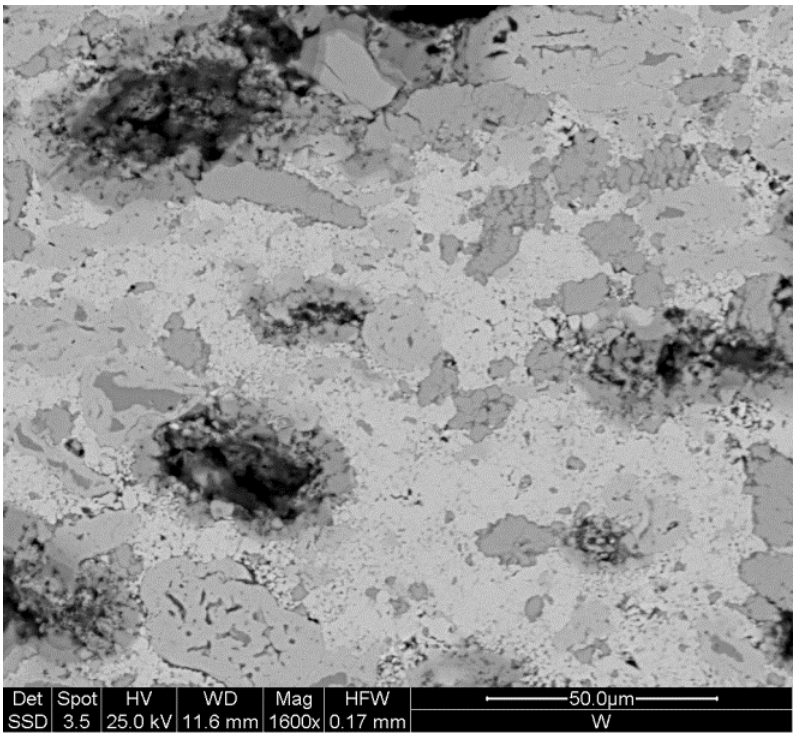


Fig. 7: FeCoNiCrAl microwave synthesized at 2.45 GHz, 300W power

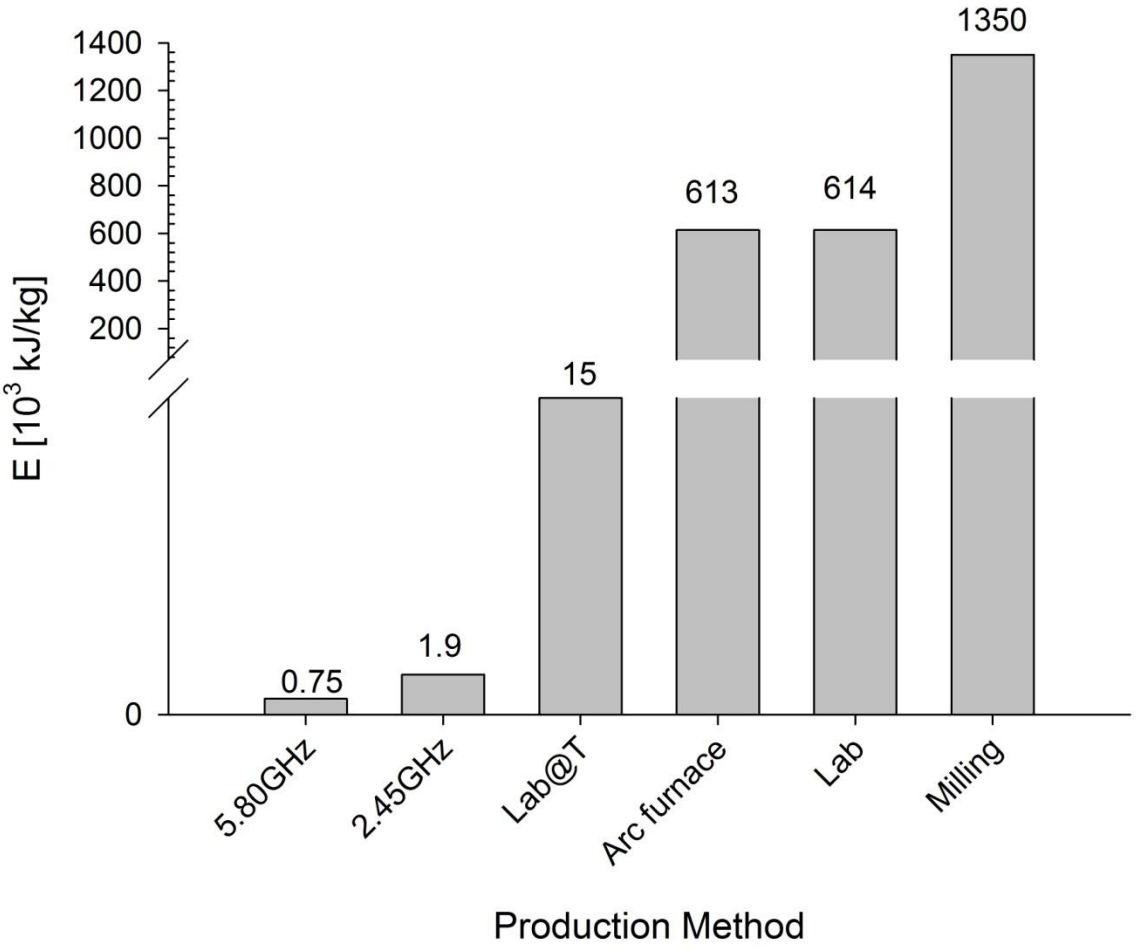


Fig. 8: Energy consumption of different technologies used to synthesize $FeCoNiCr_{0.5}Al_2$

Table 1: Suitable range to form solid solution

	ΔS_{mix}	ΔH_{mix}	δ
Solid Solution Phase	$10 < \Delta S_{mix} < 19.5$	$-22 < \Delta H_{mix} < 7$	$0 < \delta < 8.5$
BMGs	$7 < \Delta S_{mix} < 14$	$-35 < \Delta H_{mix} < -8.5$	$\delta < 9$

Table 2: Synthesis conditions

Method	**P[W]**	**Time [s]**
Microwave 5.8 GHz	180	25
Microwave 2.45 GHz	300	38
Lab Furnace @ 1100°C	1200	72
Lab Furnace including heating	1200	3072
Ball Milling	750	216000
Arc Furnace [21]	46000	80