

## Sintering of Alloyed Ni<sub>3</sub>Al Starting from Mechano-composites Powders

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### Abstract

Considering the idea that some properties, especially the mechanical properties of Ni<sub>3</sub>Al at ambient temperature can be improved by adding of some substitutional/interstitial elements, our goal was to obtain these materials starting from mechano-composites powders. In this aim, using mechanical alloying techniques three type of mechano-composite powders starting from elemental powders were obtained. Then, by reactive sintering in argon atmosphere at temperature over 900°C, alloyed Ni<sub>3</sub>Al materials were realized. This paper presents our research results regarding the microstructural aspects and phase formation in obtained materials.

**Keywords :** mechano composites powders, alloyed Ni<sub>3</sub>Al, reactive sintering, X-ray diffraction, Moessbauer spectroscopy

### 1. Introduction

Ni<sub>3</sub>Al intermetallics are of a great interest for some structural applications, especially those at the high temperature. This permanently ordered compound up to its melting point has attracted a wide interest as it is the prototype of one constituent phase of Ni-base superalloys strengthened by  $\gamma'$ -phase precipitation. The intermetallics have attractive properties: low density, high strength and good corrosion resistance, which recommend them for high temperature structural application like turbine blades or nozzles for gas burners. In spite of these attractive properties, intermetallics have limited applications due to their extreme brittleness. [1] The major cause of this brittleness is now known to be the moisture induced by environmental conditions [2].

Because high differences exists between the densities of the elements it was considered that mechanical alloying can produce a better homogenization degree of the mixes and by this, obtaining of improved properties for the resulted materials. [3]

In this paper we present our research results regarding the influence of the boron and iron adds on the microstructure and phase formations in Ni<sub>3</sub>Al compounds.

### 2. Experimental and Results

Starting from elemental powders (Ni – carbonyl, Al atomized powder with the size less than 71  $\mu\text{m}$ , Fe atomized powder with the size less than 100  $\mu\text{m}$ ) and boron powder with the size less than 50  $\mu\text{m}$ , three compositions were realized (Table 1). After three hour mechanical milling of the mixture, followed by a wet homogenization using 2% polyvinyl alcohol solution, some rectangular samples

were made (22 x 17 x h mm) by uniaxial pressing at a compacting pressure of 200 and 400 MPa. The obtained samples were synthesised in argon atmosphere.

**Table 1. The chemical composition of the experimental mixtures.**

Mixture Type	Code	(%weight)			
		Ni	Al	Fe	B
Ni <sub>3</sub> Al	Ni <sub>3</sub> Al	86.72	13.28	-	
Ni <sub>3</sub> Al-Fe-B	C2	84.93	13.91	1.11	0.05
Ni <sub>3</sub> Al-Fe-B	C5	85.5	11.8	2.7	0.05
Ni <sub>3</sub> Al-Fe-B	C6	79.35	10.6	10	0.05

The synthesis was realized using self propagation high temperature synthesis – thermo-explosion mode at 1000 °C temperatures: for two hours. The unalloyed samples, Ni<sub>3</sub>Al code, were synthesised at 650 °C temperature.

The sintered samples were then repressed in order to increase the physico-mechanical properties of the materials.

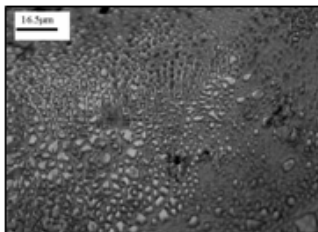
Table 2 presents the physical characteristics obtained for the synthesised materials.

Figure 1 shows the microstructural aspect after repressing of C5-1 material sample. Presence of very small grains proves the benefic effect of boron adding, too.

Quantitative micro-chemical analysis (EDX) (Table 3) shows that the synthesised materials have a high non-homogenous chemical composition and microstructure.

**Table 2. The obtained values for some physical characteristics of the synthesised materials.**

Sample code	P <sub>c</sub> tf/cm <sup>2</sup>	M <sub>c</sub> g	V <sub>c</sub> cm <sup>3</sup>	ρ <sub>c</sub> g/cm <sup>3</sup>	V <sub>s</sub> cm <sup>3</sup>	ρ <sub>s</sub> g/cm <sup>3</sup>	ρ <sub>r</sub>	HB <sub>s</sub>	HB <sub>r</sub>
C2-1	2	8.44	1.8722	4.5080	1.774	4.746	5,9	91.8	127
C2-2	2	7.88	1.7624	4.4711	-	-	-	-	-
C2-3	4	7.96	1.5794	5.0399	1.428	5.566	6,08	124.0	162
C2-4	4	7.62	1.5046	5.0643	-	-	-	-	-
C5-1	2	8.07	1.7569	4.5932	1.837	4.392	-	93.9	137
C5-2	2	8.04	1.7393	4.6226	-	-	-	-	-
C5-3	4	7.77	1.4899	5.2153	1.548	5.005	5,97	93.9	145
C5-4	4	7.82	1.4955	5.2291	-	-	-	-	-
C6-1	2	8.03	1.7024	4.7170	1.648	4.866	5,77	83.9	127
C6-2	2	7.89	1.6754	4.7092	-	-	-	-	-
C6-3	4	8.18	1.5372	5.3215	1.576	5.19	6,15	79.1	134
C6-4	4	8.1	1.5230	5.3186	-	-	-	-	-



**Fig. 1. C5-1 material–25 tf repressed.**

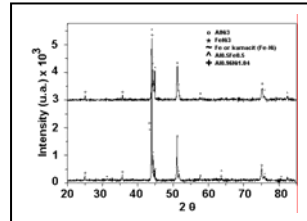
**Table 3 Quantitative chemical microanalysis (EDX).**

Material	Fe		Ni		Al	
	%at	%wt	%at	%wt	%at	%wt
C2-3-s	1,12	1.147	85,93	92.43	12,9	6.41
C5-1 -r	2,53	2.483	92,15	94.98	5,32	2.52
C5-1 -r	2,55	2.51	91,36	94.58	6,09	2.9
C5-3 - s	3,49	3.5	87,31	92.03	9,20	4.46
C5-3 - s	3,60	3.56	89,82	93.29	6,58	3.14
C6-3 - s	9,86	10.55	70,61	79.34	19,5	10.1
C6-3 - s	10,46	10.47	81,32	85.54	8,22	3.98

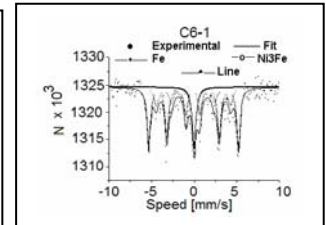
Note s – as sintered; r – as repressed

In the case of C2 composition it can be observed that the Ni and Fe are uniformly distributed into the matrix, while the Al are presented in agglomerated form intragranular and at grain boundary. It means that the homogenization degree of the mixture was not optimum. The tested fields of C5 composition show that the material is completely un-homogenous. The Fe is still found as a reactant while the Al is distributed especially in the proximity of pores. The presence of Al near the Fe (C6 composition) proves that the temperature synthesis must be higher. C5 Material in repressed form, have uniform distributed Al and Ni. The Fe is in agglomerated form which not contributes at the diffusion/reaction processes. Only a small quantity of Fe is uniform distributed in the matrix, fact which is proved by X-ray analysis (Fig.2) and Moessbauer spectroscopy (Fig.3), too. The X-ray diffraction show that the Ni<sub>3</sub>Al with disordered cubic crystalline lattice is the majoritary phase having a expanded lattice parameter with a<sub>0</sub> = 3,57 Å. Near

this majoritary phase, co-exists the Fe<sub>3</sub>Ni having a izomorph crystalline lattice with those of Ni<sub>3</sub>Al. Also, X-ray diffraction evidenced forming of Al<sub>0.5</sub>Fe<sub>0.5</sub> and Al<sub>0.96</sub>Ni<sub>1.04</sub> minority phases and the presence of a small quantity of Fe un-reacted .



**Fig. 2. C6 material- X-ray Diffraction**



**Fig. 3. C6- Moessbauer spectrum**

The Mössbauer measurements performed on the obtained materials showed that the site occupation of Fe atoms depends on Fe concentration.

### 3. Summary

The mechanical milling of the mixes for three hours is insufficient for obtaining of mechano-composite. The synthesis temperature must be higher to obtain the necessary heat in the system so that the combustion synthesis to be not perturbed. The diffraction analyses clearly show that the iron alloyed Ni<sub>3</sub>Al is not formed. At the adopted temperature synthesis, only iron alloyed NiAl was obtained. Anyway, the obtained contracted lattice parameter shows that the Ni<sub>3</sub>Al phase is not pure.

The Mössbauer technique shows that the iron distribution depends on the temperature synthesis, matrix composition and on the iron concentration.

### 4. References

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