Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych

20: 1 (2020) 7-10

COMPOSITES THEORY AND PRACTICE
ISSN: 2084-6096
ISSN (online): 2299-128X

Maciej Szlezynger[1], Łukasz Maj[1,*], Małgorzata Pomorska[1], Jerzy Morgiel[1], Katarzyna Jach[2], Marcin Rosinski[2]

[1] Institute of Metallurgy and Materials Science, Polish Academy of Sciences,ul. W. Reymonta 25, 30-059 Krakow, Poland
[2] GeniCore, ul. Wólczyńska 133, 01-919 Warsaw, Poland
**Corresponding author. E-mail: l.maj@imim.pl*

Received (Otrzymano) 10.01.2020

EFFECT OF UPGRADED FIELD ASSISTED SINTERING TECHNOLOGY ON MICROSTRUCTURE OF NiAl/CrB_2 COMPOSITES

The method of fabricating metal matrix composites plays a crucial role in obtaining dense materials characterized by high wear resistance. The present work describes an attempt to produce NiAl/CrB_2 composites using the next-generation spark plasma sintering (SPS) method, i.e. upgraded field assisted sintering technology (U-FAST) technique. Microstructure characterization was performed by means of scanning (SEM) and transmission (TEM) electron microscopy. The SEM microstructure investigations of the NiAl model material proved practically full densification of the material sintered at 1200°C and 1300°C, even if remnants of surplus nickel were observed at the boundaries of rounded NiAl grains. The NiAl/CrB_2 composites, besides fused NiAl and CrB_2 grains, showed the presence of a raised level of nickel also at the grain boundaries. The TEM microstructure observations helped to establish that even if the grain boundaries were pinned by nickel-rich precipitates, some increase in grain growth took place, as evidenced by the fact that strings of smaller precipitates were also visible outside the matrix grain boundaries. All these microstructure investigations indicate that the newly elaborated U-FAST technique is evidently capable of producing compacts free of porosity at lower temperatures and during a shorter time than solid hot pressing or vacuum sintering in a semi-liquid state.

Keywords: metal matrix composites, SPS, microstructure, electron microscopy

INTRODUCTION

Wear resistant metal matrix composites (MMC) are an extension of Babbitt's XIX century idea of a bearing material based on a soft alloy with interspersed hard particles of an intermetallic phase. Substituting the latter with much harder ceramic ones, like borides, makes sense only if the matrix is able to give them proper support, what caused the intermetallic phase to be graduated to the role of the matrix. This approach simultaneously requires establishing very good binding between the matrix and the hard particles, difficult to achieve in the case when they belong to different material groups.

Intermetallics, like NiAl or TiAl, are well known for both their high hardness and oxidation resistance, promoting them to the position of an excellent MMC matrix. At the same time, they are susceptible to abrasive wear at higher temperatures which, however, may be alleviated through the addition of ceramic strengthening phases. In the case of plasma sprayed MMC coatings with an NiAl matrix, a number of diboride phases like TiB_2, ZrB_2 or CrB_2 have already been used [1]. Among them, the highest improvement in wear resistance was obtained with an addition of 15 wt.% CrB_2 phase [2]. The above progress was to a large extent achieved due to the plasma spraying method, which is realized by at least partial melting of the deposited material, ensuring good binding. The accompanying experiments aimed at obtaining bulk MMC using hot pressing (performed in the temperature range for which the material is in solid state) or vacuum sintering (with a partly melted matrix) produced not only coarse-grained but also quite porous (~5%) material, making it too brittle for most practical applications aside of crushing for powder plasma spraying [3].

Spark plasma sintering (SPS), also known as the field assisted sintering technology (FAST) technique, bears some similarities to plasma spraying as the short DC current pulses applied to the compacted particles result in local discharges raising the temperature in these areas up to 10^3 K [4-6]. The discharges remove the native oxides and open the way for mixing of the surface of the melted materials establishing a strong bond between the involved particles. Many mechanisms of the SPS/FAST process enhancing sinterability are considered, among others: Joule heating, field assisted sintering, local melting as well as surface cleaning of the powders to be sintered. The above approach allows not only the sintering temperature to be lowered by 200÷300ºC with respect to conventional hot pressing, but also makes the joining of particles presenting highly dissimilar melting temperatures easier, like that of an intermetallic matrix and ceramic particles. The joining of the particles stems from the fact that locally the tem-

perature exceeds both of them. This technique was successfully applied to iron-based [4], alumina [5] or MMC [6] powders, but proof of direct fusion of the ceramic and metal in composites obtained employing this technique is still lacking.

The main aim of the present work was to investigate the effect of sintering using the upgraded FAST (U-FAST) method, i.e. the next generation of SPS working with shorter pulses of higher voltage, on the microstructure of an NiAl/CrB_2 composite material. Applying a higher voltage in the U-FAST process creates specific conditions to promote plasma cleaning of the particle surface. U-FAST is a remarkable technique for consolidating a wide range of materials [5]. The task involved thorough microstructure characterization carried out by means of scanning (SEM) and transmission (TEM) electron microscopy methods.

MATERIALS AND METHODS

In the present work, nickel aluminide (99.9% purity/45 μm average size) and chromium diboride (99.9% purity/40 μm average size) powders were used. They were obtained by courtesy of Goodfellow and Polema JSC companies, respectively. The model NiAl powder as well as an NiAl+15 wt.% CrB_2 mixture were subjected to milling by a Fritsch Pulverisette 6 planetary mill equipped with zirconia balls, at a rotation speed of 200 rpm for 8 h, and then sieved to a powder size of less than ~20 μm. The NiAl model material and the composite containing an NiAl intermetallic phase with the addition of a 15 wt.% CrB_2 phase were sintered in vacuum (~10^{-5} mbar) using the U-FAST GeniCore High Vacuum system model GC_C_V0L3HV - 350 kN, 100 kW. 14V DC voltage was applied in pulses lasting ~1 ms with ~1 ms breaks. The consolidation process of each sample consisted of two stages as described in detail in Tables 1 and 2.

TABLE 1. **Parameters of consolidation of NiAl powders**

Sample	Stage	Pressure [MPa]	Heating rate [°C/min]	Temperature [°C]	Time [min]
IM_01.002	1	20	50	600	3
	2	100	50	1200	15
IM_01.003	1	20	50	600	3
	2	100	100	1300	15

TABLE 2. **Parameters of consolidation of NiAl/CrB_2 powders**

Sample	Stage	Pressure [MPa]	Heating rate [°C/min]	Temperature [°C]	Time [min]
IM_02.001	1	20	50	600	3
	2	100	50	1200	10
IM_02.002	1	20	50	600	3
	2	100	100	1300	10

Microstructure observations in the microscale were carried out with a Philips XL30 scanning electron microscope operating at 18 kV in the backscattered electrons (SEM/BSE) mode. They were performed in order to examine the microstructure of the pure NiAl sinters and the distribution of chromium diboride particles in the NiAl matrix for the NiAl/CrB_2 composite material.

Nanoscale characterization of the obtained materials was performed with the help of an FEI Tecnai G^2 200 kV FEG transmission electron microscope. The microstructure observations were carried out in the bright field mode (TEM/BF). The thin foils for TEM observations were cut out with Ga+ ions using a FEI Quanta 3D 200 Dual Beam Focused Ion Beam microscope equipped with an Omniprobe lift-out system.

RESULTS

The SEM/BSE observations of polished sections of the NiAl model material sintered by the U-FAST method at 1200 and 1300°C showed that the grains formed during milling were fully densified, producing a material practically free from porosity (Fig. 1). The average grain size stayed the same during this treatment for both the applied temperatures. The presence of white particles at the grain boundaries suggests that the nickel content in the starting material slightly exceeded the solubility limit (i.e. ~65 at.% observed in a binary phase diagram) for the NiAl intermetallic. The white particles were occasionally accompanied by dark features, which might represent either small pores or oxides.

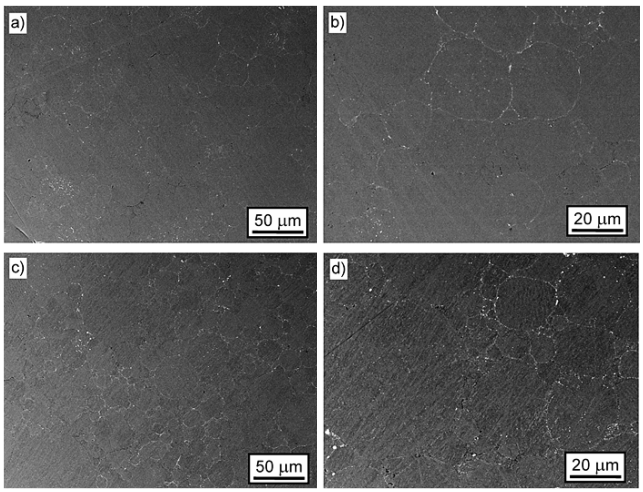

Fig. 1. SEM/BSE microstructure micrographs of NiAl compacts sintered by U-FAST method at 1200°C (a, b) and 1300°C (c, d)

Similar SEM/BSE observations of the NiAl/CrB_2 composites, sintered in the same pressure-temperature-time conditions, indicated that despite the presence of ceramic particles, the size of the NiAl grains is comparable to those found in the NiAl model material (Fig. 2). However, the composite obtained at the lower temperature shows the occasional presence of layered metallic/ceramic structures (encircled with a dashed line in

Fig. 2a), while none of them were present in the material sintered at the higher temperature. Generally, the latter was more homogenous with an evidently smaller amount of fine grains than in the former. The micrographs taken at higher magnifications showed the presence of nickel veins also inside the NiAl grains. Most of the ceramic particles were distributed at the NiAl grain boundaries. On average, they were smaller than the NiAl grains, but occasionally larger ones were also noted.

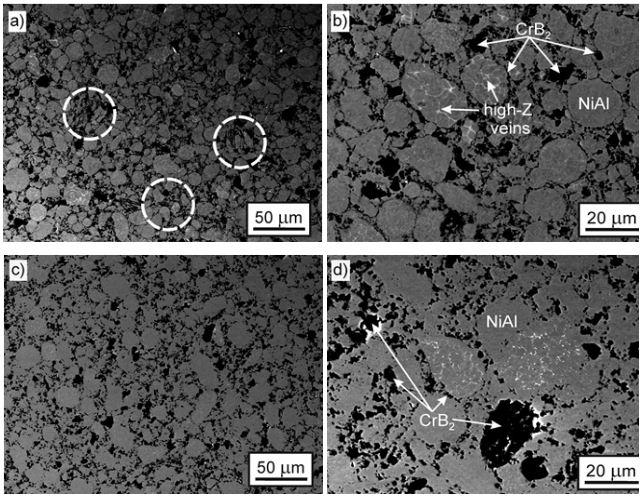

Fig. 2. SEM/BSE microstructure micrographs of NiAl/CrB_2 composites sintered by U-FAST method at 1200°C (a, b) and 1300°C (c, d)

The TEM/BF observations of the NiAl model material helped to establish that aside of larger precipitates at the grain boundaries identified by SEM/BSE, others pinning the grain boundaries within the grains were also visible (Fig. 3a). Regardless of the above, limited grain growth evidently took place as some of the smaller precipitates were by-passed by the grain boundaries, leaving behind a string of small crystallites (Fig. 3b).

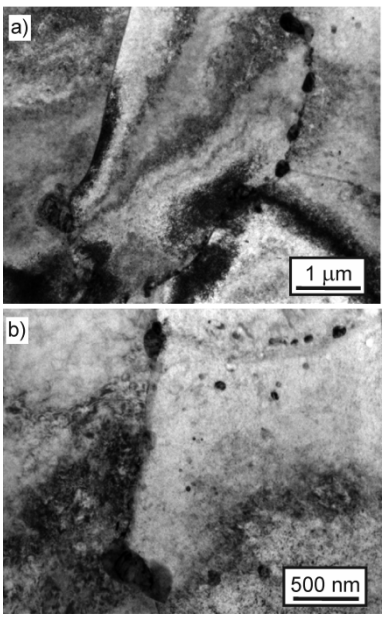

Fig. 3. TEM/BSE microstructure micrographs of NiAl sintered by U-FAST method at 1200°C presenting precipitates (a) at grain boundaries and (b) within grain interior

Within the NiAl/CrB_2 sinters, the grain boundaries were predominantly populated with numerous boride particles (Fig. 4). Even if the latter frequently remained in direct contact, they show very good wetting with the matrix, i.e. no porosity was encountered within the investigated areas.

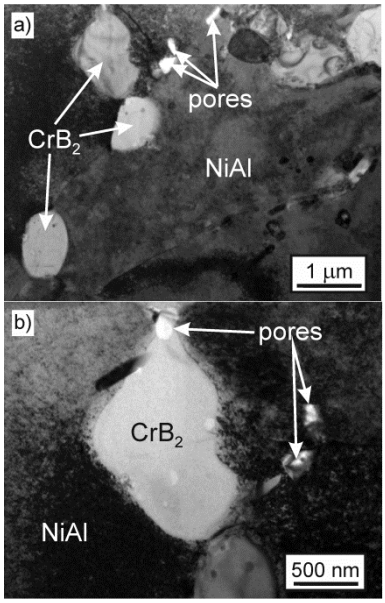

Fig. 4. TEM/BSE microstructure micrographs of NiAl/CrB_2 composite sintered by U-FAST method at 1200°C presenting borides and oxides (small, elongated) at grain boundaries taken at (a) lower and (b) higher magnifications

SUMMARY

The performed experiments proved that by means of the U-FAST method it is possible to produce a dense NiAl intermetallic material as well as NiAl/CrB_2 composites. It should be noted that even in the latter case all the particles, i.e. metallic and ceramic, were fully fused. By adhering to the process parameters in the shorter time - lower temperature windows, it was possible to retain a fine-grained matrix. Both of them give a perspective of decreasing wear, even in conditions involving simultaneous high loading at raised temperatures.

Acknowledgements

The research was supported by the National Science Centre (NCN) of Poland within research project No. 2018/29/N/ST8/02427 entitled "Elaboration of new method of NiAl(CrB_2) composite coatings deposition by mechanical embedding of powders into metallic substrates".

REFERENCES

[1] Umanskyi P., Poliarus O., Ukrainets M., Martsenyuk I., Effect of ZrB_2, CrB_2 and TiB_2 additives on the tribological characteristics of NiAl-Based gas-thermal coatings, Key Engineering Materials 2014, 604, 20-23.

[2] Umanskyi P., Brodnikovskyi M.P., Ukrainets M.S., Poliarus O., Stelmakh O.U., Brodnikovskyi D.M., Effect of chromium di-boride additives on strength properties of NiAl-CrB_2 composites in a wide temperature range, Powder Metallurgy and Metal Ceramics 2018, 56, 681-687.

[3] Poliarus O., Morgiel J., Umanskyi O., Szlezynger M., Pomorska M., Bobrowski P., M. Szczerba, Microstructure and phase composition of NiAl-CrB_2 composite powders used for plasma spraying, Composites: Theory and Practice 2018, 18, 121-124.

[4] Hebda M., Spark Plasma Sintering - Nowa technologia konsolidacji materiałów proszkowych (in Polish), Mechanika/ Mechanics 2012, 109, 47-55.

[5] Jach K., Rosiński M., Translucent aluminium oxide ceramics manufactured in U-FAST technology, Ceramic Materials 2018, 70, 4, 293-300.

[6] Schembri J., Kaur S., Oskouei R.H., Rosinski M., Ghomashchi R., Rumian R., Understanding the potential of U-FAST sintering process - an overview, Aspects in Mining and Mineral Science 2019, 3, 367-372.

