Journal of Achievements in Materials and Manufacturing Engineering
VOLUME 17
ISSUE 1-2
July-August
2006

Influence of the chemical composition and particle size of the metal matrix, on TiCN-reinforced Fe-based composites

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Received 15.03.2006; accepted in revised form 30.04.2006

Materials

ABSTRACT

Purpose: The objective of this work is to study the influence of different parameters as the chemical composition and particle size of the metal matrix, on TiCN-reinforced Fe-based composites.

Design/methodology/approach: In order to obtain the composite powder two different types of blending were used, conventional blending and high energy milling (HEM). The HEM was carried out in a planetary ball mill during 12 hours, with a rotating speed of 400 rpm, and a ratio ball:powder of 10:1 (in mass). The atmosphere was Argon to prevent the oxidation. After the preparation of powders, compacts were produced by uniaxial pressing at 700 MPa and sintering under vacuum. The sintering temperatures varied between 1350ºC and 1450ºC, for 60 min. Sintered samples were characterised by determination of density, dimensional change, Vickers hardness (HV30), bending strength, and C, N contents (by LECO). The microstructural study was carried out by scanning electron microscopy (SEM).

Findings: As a result of the study it is clear that the presence of carbides in the metal matrix allows the increasing of mechanical properties of sintered composites, and these properties are related with the microstructure and C/N ratio.

Practical implications: In this research 50 % vol of hard phase is introduced, following a simpler and lower-cost route, as pressing and sintering. It is true that high-energy milling raises the cost of the processing. This is why conventional blending of small size powder particle has also been done in this work. The latter route has shown to give quite promising results, reaching hardness values about 2000 HV30.

Originality/value: In this work, composite materials with high hardness have been obtained following a simple and low-cost route.

Keywords: Metal matrix composites; TiCN; Mechanical milling

1. Introduction

Titanium carbonitride (TiCN) based cermets were developed from TiC based cermets by the addition of TiN in the 1970s [1]. Because of their high hardness, thermal stability, relatively high thermal and electrical conductivities, and excellent creep and wear resistance, cermets have been improved the performance of cutting tool made of WC-based cemented carbide [2]. They have been successfully utilized for cutting tool in semi-finishing and finishing work on steel and cast iron [3]. Modern cermets are normally made of TiCN solid solution or mixtures of TiN and TiC as main hard component and Co/Ni as binder. However, these elements are scarce, expensive, and their dust is especially harmful. This leads to an interest for replace this matrixes total or partially for other harmless elements like Fe. However, some difficulties have been found for the introduction of hard particles in a Fe matrix, the main of which are agglomeration of particles that do not permit an homogeneous dispersion of hard phase into the matrix, and the poor bonding between added particles and the matrix [3]. One of the techniques proposed for avoiding or diminishing these problems is high-energy milling [4,5], which permits to introduce high percentages of hard phase [6]. Extensive research from the literature indicates that properties of TiCN-based cermets are intensively dependent on their chemical compositions and microstructure [7,8]. In this paper the effect of HEM on the properties of Fe-based composite is studied, also the effect on the microstructure of the alloying elements such as Cr is studied.

2. Experimental process

In order to study the effect of the matrix composition only one type of reinforcing particles was used, titanium carbonitride (TiCN), with a percentage of addition of 50% vol. Three different steels were used as the matrix: Fe - 0.25 % wt C, Fe -15 % wt Cr - 0.25 % wt C, and high speed steel, grade M2. In order to obtain the composite two different types of blending were used, conventional blending and high energy milling (HEM). The HEM was carried out in a planetary ball mill during 12 hours, with a rotating speed of 400 rpm, and a ratio ball:powder of 10:1 (in mass). The atmosphere was Argon to prevent the oxidation. Table 1 shows the type of matrix, the type of blending used and the particle size after blending for each composite. Note that in all the cases the percentage of reinforcement, TiCN, is 50 % vol.

Table 1.
Characteristics of powder matrixes and blending

Matrix Powder	Name	Type of Blending	Particle size (μm) after blending
Fe (ASC 100-29, Höganäs, Sw)	Fe1	HEM	15
Fe Carbonyl (Ecka granules. GmbH)	Fe2	Conventional	12-25
Fe (carbonyl or ASC??)-15Cr	Fe-Cr	HEM	23
M2 (Osprey Ltd.)	M2	Conventional	22

After the preparation of powders, compacts were produced by uniaxial pressing at 700 MPa and sintering under vacuum. The sintering temperatures varied between 1350ºC and 1450ºC, for 60 min and 120 min. Sintered samples were characterised by determination of density, dimensional change, Vickers hardness (HV30), bending strength, and C, N contents (by LECO). The microstructural study was carried out by scanning electron microscopy (SEM).

3. Results

3.1. Effect of chemical composition

The addition of Cr for the lowest sintering temperature (1350ºC) increases both density and shrinkage with respect to the plain Fe matrix. When temperature increases, the values are similar for the two matrixes considered and, for the highest temperature (1450 ºC) the Fe-Cr matrix led to the melting of the material. The addition of Cr to the Fe matrix was made after the reported benefits found in the literature, that indicated an improvement in the wettability of the Fe-rich liquid phase formed during sintering on the TiCN [9], when 15 % Cr was added. In our case, the benefits on density seem to be less important from 1400 ºC, which could be due to the elimination of Cr from the system during the vacuum sintering, leading to less improvement in wettability than expected. However, the values of hardness are slightly improved in Fe-Cr matrix materials with respect to plain Fe matrix (Fe1), as it can be seen in figures 1. For 1350ºC, hardness are the same, but for 1400 ºC, hardness improve even though the density is the same. This can confirm a better interaction between matrix and reinforcement in materials with addition of Cr. In any case, values of hardness are quite low for both materials, and this is why other alternatives had to be found. The effect of Cr in sintered properties can also be seen in values of bending strength (figures 2). Both for 1350 ºC and for 1400 ºC, the Fe-Cr matrix provides higher strength, being much significant for the latter. This behaviour could again be interpreted as an improvement of the interaction between metal matrix and ceramic particles due to the addition of Cr.

As results from high-energy milled Fe-based composites were lower than expected, two other powders where chosen for the matrix: an Fe-carbonyl powder and an M2 grade high-speed steel, both with smaller particle size than previous Fe powders, to avoid the HEM process. In this section the discussion is focused in the composition of the matrixes, that is, the effect of the alloying elements on the properties of sintered composites.

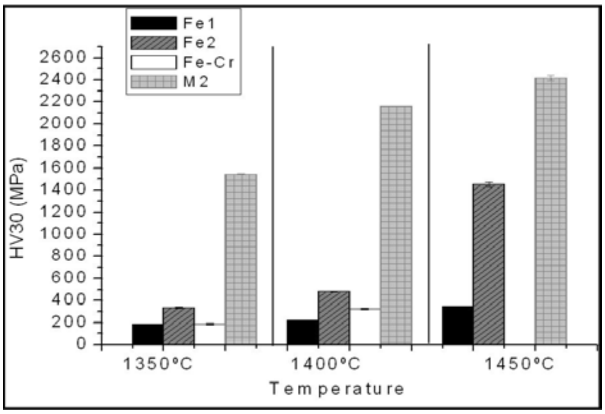

Fig. 1. Variation of hardness (HV30) with temperature for 1 hour of sintering

Regarding the hardness, it is with noticed that hardness of Fe2 material (the one from Fe-carbonyl powder) presents higher values that Fe1 (from HEM), for both sintering times (see figures 1). The highest values of hardness (about 1400 HV30, equivalent approximately to 74 HRC) were obtained for materials sintered for 1 h at 1450 ºC. This is a remarkable result, as this material is in the as-sintered condition, and the matrix is Fe-C. For 2 h of sintering time, this result was not achieved, probably due to the grain growth [8]. The same effect of the sintering time can be observed on hardness values of M2 materials, which drop from 2400 HV30 when sintered at 1450 ºC for 1 h, to about 1200 HV30 when sintering time is 2 h for the same temperature. This results for M2 materials are again remarkable, as they are higher than any composite of these characteristics, and even higher that cemented carbides[10] **.**The explanation lies in the composition of M2 used as matrix. This steel contains high percentage of carbide former elements, such as Mo, W, and V, and also contain 4 % Cr, that can contribute to the better wettability, as mentioned before. Then, the final composite contains higher percentage of hard phase, and also, the presence of alloying elements improve interactions between metal and added ceramic phases, that allows that relative density is close to full density . In spite of the high hardness of M2-based materials, the bending strength is lower than expected, especially for 1450 ºC, where values are the same for the Fe-carbonyl based material and for the M2 one. In general, the bending strength values of all the materials studied are low, and lower than other composites [11].

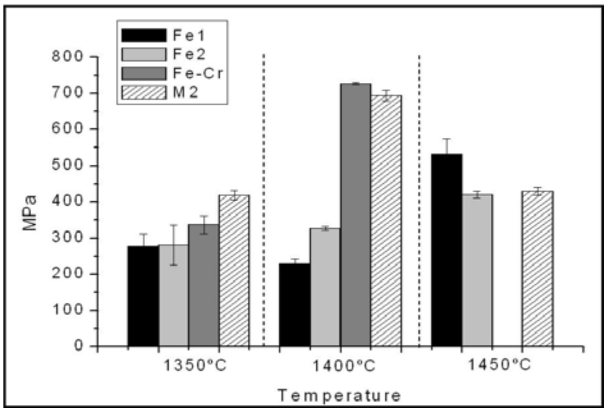

Fig. 2. Variation of the bending strength with temperature after 1 hour of sintering

Fracture surface was examined in order to identify the failure initiating sites and crack propagation modes. Spherical holes have also been observed in the fracture, these pore-like cavities have been previously reported as failure initiation sites [12].The origin of these pores is probably related to the agglomeration of lubricant.

3.2. Effect of particle size of metal matrix

The main objective of High Energy Milling (HEM) is obtain an homogeneous distribution of the hard phase into the more ductile particles of the base powder to obtaine a "composite powder" and also, to reduce the particle size of the initial powder (Table 1) [4,6,13]. However, milling process promotes changes in chemical composition of the powder [14]. During 12h of milling different reactions take place; inside the mill oxygen is present and it reacts with the carbon, leading to the formation of gases such as CO y CO_2. Moreover, during sintering process the chemical composition also changes. The reason is that processes such as TiN decomposition, take place, and these processes are beneficiated with high temperature [15].

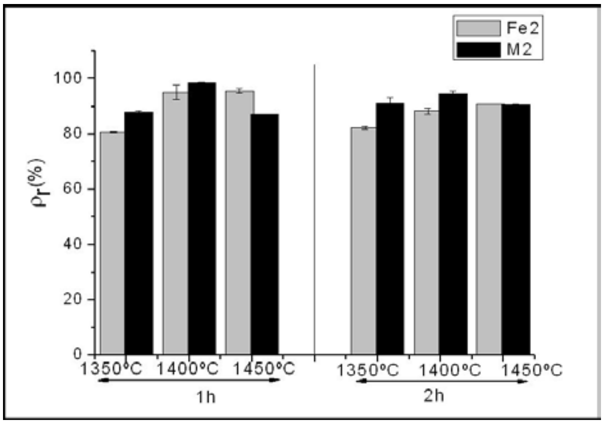

Fig. 3. Variation of relative density with sintering temperature and time, for Fe-carbonyl (Fe2) and M2 based materials

To avoid these modifications that can influence the sintering behaviour and the final properties of the composites, powders with small particle size where chosen to be processed in the same conditions than HEM powders, after blending in a conventional way (using a turbula for 4 hours). Figure 3 shows the relative density of materials made from Fe-carbonyl and M2 powders as matrix, which can be compared with values of HEM powders in figure 1. In general, values of density are higher for these powders than for HEM powders, probably due to the higher green density that the former.Some authors reported [16] that the wetting characteristics of a liquid phase formed during the sintering are influenced by N content in TiCN, decreasing the wettability of the liquid phase when N content decreases. This is the possible reason why mechanical properties of Fe1 and Fe2 are different in spite of the similar composition and particle size (Table 1) of the base powder.

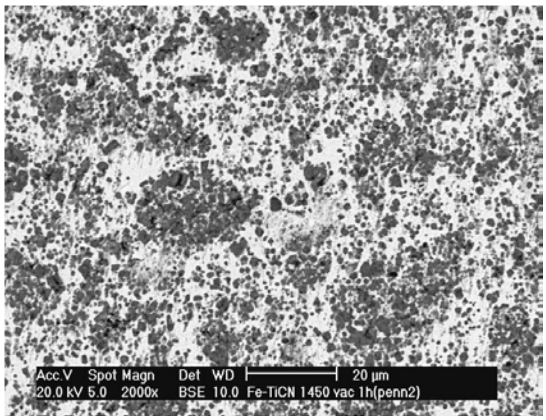

Fig. 4. Microstructure of Fe1 sintered at 1450ºC for 1 hour

Finally, microstructures of materials studied are shown. The phases distribution in Fe1 and Fe2 are similar despite the latter was obtained with conventional blending (Fig. 4 and 5). The main difference is the size of the ceramic phase (dark contrast), being smaller for HEM process, but also more concentrated in some areas, where previous composite particles where placed. Figure 6 shows M2 microstructure, in this case two different types of carbides are present: the bright contrast phase is constituted by carbides from alloying elements of the M2 high-speed steel, while dark contrast phase corresponds to TiCN. It can be seen that bright carbides appear surrounding the TiCN particles, allowing a better bonding between TiCN particles and metal matrix.

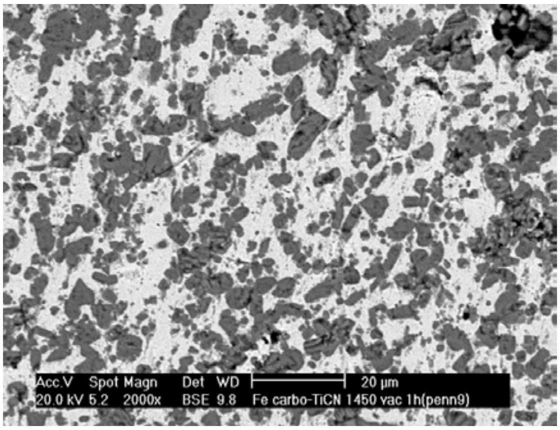

Fig.5. Microstructure of Fe2 sintered at 1450ºC for 1 hour

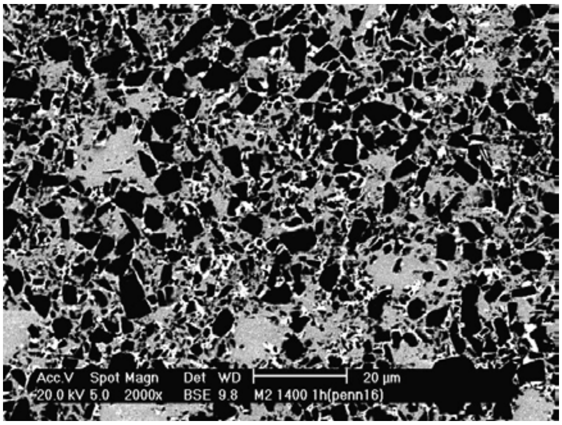

Fig.6. Microstructure of M2 sintered at 1400ºC for 1 hour

4. Conclusions

The effect of the chemical composition and particle size of the metal matrix on the properties of Fe-based composites has been studied. Cr addition increase properties such as hardness and bending strength, the reason is Cr improves the wettability and, therefore, the interaction between ceramic and metal phase. But the sintering conditions, in the Fe-Cr system, promote to loss of Cr below 15 wt% and mechanical properties are not as higher as expected. On the order hand, it has confirmed that using M2 high speed steel as metal matrix increases the mechanical properties, obtaining relative density values close to full density. The tests to evaluate the effect of particle size, shows that the high-energy milling does not increase the mechanical properties, this is due to the chemical composition, that does not remain constant during the milling process, where losing of carbon and nitrogen were found. This composition also changes during sintering process. The material obtained with conventional blending present better properties but the bending strength is not similar than other composites.

Acknowledgements

The authors wants to acknowledge Center for Innovative Sintered Products (CISP) and Penn state University for the use of equipments needed to develop this study, and to Prof. R.M. German for his supervision. Thanks to Fundación Universidad Carlos III de Madrid, Instituto "Álvaro Alonso Barba" The CICYT through the R+D project MAT2003-03376 and the MEC through the project DPI2005-08018, for the financial support.

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