8º Congresso Nacional de Mecânica Experimental
Guimarães, 21-23 de Abril, 2010

OPTIMIZATION OF SINTERING TEMPERATURE AND COMPACTION PRESSURE OF STAINLESS STEEL/SIC COMPOSITES

Carvalho, O,[1], Soares, D.[1], Silva, F.S.[1]

[1] Centre for Mechanical and Materials Technologies, Mechanical Engineering Department, Minho University

ABSTRACT

In this work different stainless steel composites reinforced with SiC particles were produced by the powder metallurgy technique. Different compaction pressures and sintering temperatures were used in order to study the influence of these parameters on the MMC mechanical properties. The obtained shear strength results changes with the compacting pressure as well as with the sintering temperature. The obtained composites present an interface between the reinforcement particles and the matrix. The interface reaction was characterized by SEM/EDS analysis. The effect of the sintering temperature on the type and extension of the matrix/particle interface reaction was evaluated.

1 - INTRODUCTION

Metal matrix composites, internationally known as MMCs represent a class of materials that can be used to solve complex problems related with aerospace, military, sports, biomedical, electrical transport and electronic components technologies. A great interest has emerged around MMCs due to several intersting mechanical properties such as high elastic modulus combined with a light weight (Yu, and Lee 2000). Primary manufacturing processes of MMCs are often divided in liquid solid state processes. The liquid phase processes include gravity casting; inject casting, casting pressure-assisted infiltration, pressure-assisted, semi-solid casting, spray deposition and reactive infiltration processes. The solid-state processes include powder metallurgy in which a powder mixture is followed by consolidation process (Amaral Fortes and Ferreira 2003.).

The strength properties of a MMC critically depend on the extent of interface reactions between the reinforcement and the matrix (Abenojar, et al. 2002). Systems in which interfacial reactions occur at some stage of fabrication or while exposed to temperature as a finished product are usually thermodynamically unstable, with renders their use questionable (Pelleg 1999).

One of the problems when it is used SiC as the reinforcement is the solid state reaction between SiC particles and the metal matrix which results in a

microstructure change and related properties degradation of the material as a whole (Tang et al 2002)

In this work, it will be study the mechanical properties and the chemical reactions of a MMC, with a stainless steel martensitic 410 L matrix and SiC particles as reinforcement.

2 – EXPERIMENTAL PROCEDURE

A stainless steel/SiC composite was produced by the powder technique. A martensitic 410L stainless steel powder was used 410L with a particle distribution of: 45% lower than 45 µm and 1% above 150 µm (Hoganas AB-Sweden). The alloy chemical composition is presented in table 1. The Stainless steel powder is composed also with a 1 wt% lubricant (Acrawax). A SiC powder was used as reinforcement with a mean particle size of 118 µm.

Table 1 – Stainless steel chemical composition (in wt%) **Erro! A origem da referência não foi encontrada.**.

Element	C	Cr	Si	O	N	Fe
(wt%)	0.02	13	0.8	0.24	0.03	rest

Powder mixtures of MMC, containing 10 % SiC by weight, were produced. The mixing of the powders was made inside a close polymer bottle. This bottle was placed in a rotation machine and the mixing was made with a constant rotation speed of 40 rpm during 6 days.

Compaction of the composites was made on a steel cylindrical matrix with an internal diameter of 10 mm. Three types of samples were produced by the use of an unidirectional load of 374 MPa, 749 MPa and 1123 MPa. The samples dimensions, after cold pressing, were: 10 mm in diameter and 9.36±0.82 mm of length.

The lubricant debinding was done at 550 ºC for 60 min in a closed system with a constant flow of argon. Samples were then sintered in a resistance furnace, under vacuum (~10-2mBar), with a stage of 1 h at the maximum temperature (Fig. 1).The stage temperatures used were 900, 1000, 1100, and 1180 ºC.

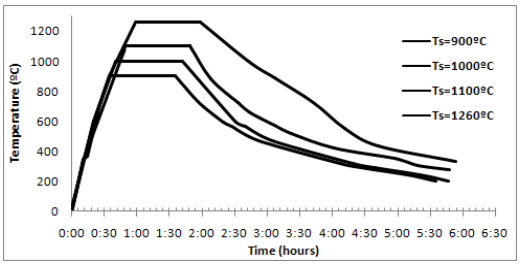


Fig. 1 – Thermal cycle used for samples sintering at the different temperature stages.

The samples final microstructure was characterized by optical (OM) and electronic scanning microscopy (SEM/EDS).

The sintered samples densities' was evaluated by the Archimedes principle method. The samples shear tests were made using the equipment presented in Fig. 2.

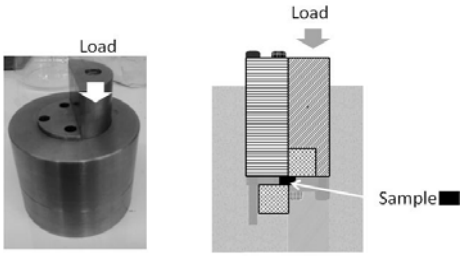


Fig. 2 – Experimental device used on the shear tests.

3. RESULTS AND DISCUSSION

The samples density was evaluated after the cold pressing and sintering operations. The results are presented in Fig. 3. The increase on the cold compaction pressure increases the final density. With the increase in the sintering temperature there is an improvement of density but less pronounced when compared with the effect of the cold compacting pressure.

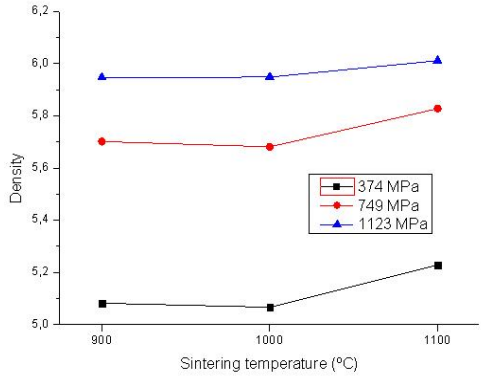
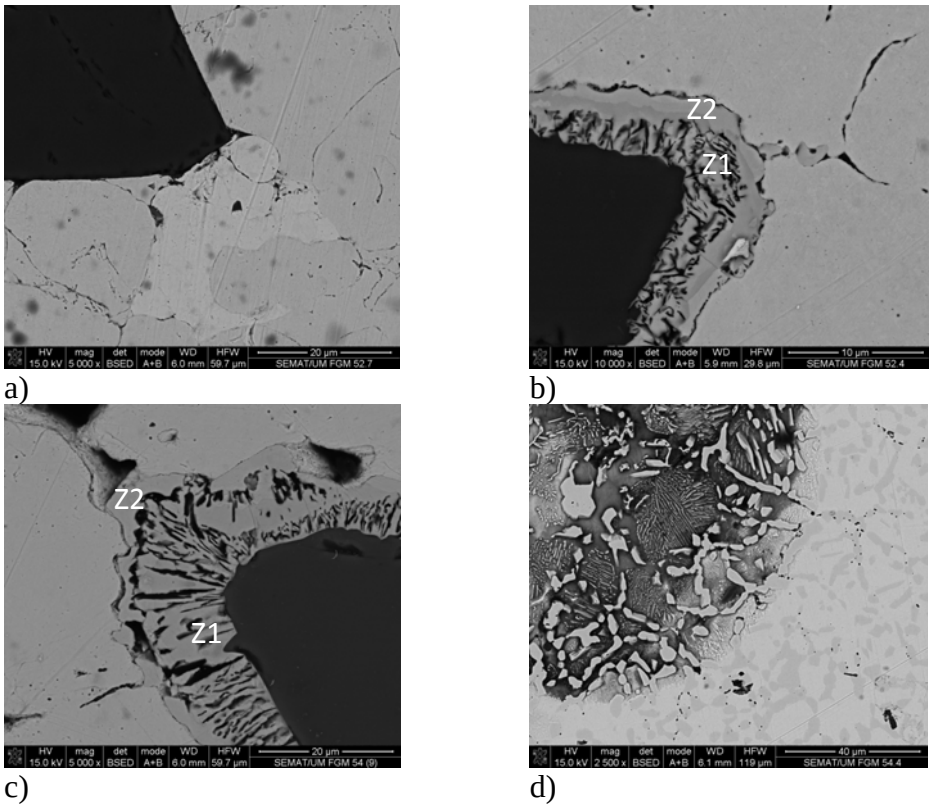


Fig. 3 – Obtained density results (Archimedes method) with different compaction pressures and sintering temperatures.

The reactivity between the metal alloy and SiC particles reinforcement was verified to be highly dependent on the sintering temperature. The interface morphology is presented in Fig. 4 for the three tested temperatures. It is shown that, for the stage time of 1 hour, there is a particle dissolution reaction for sintering temperatures above 1000ºC. Fig. 4 shows that there is a significant interface thickness increase when the sintering temperature increases from 1000 to 1100 ºC. A complete particle dissolution was obtained for samples sintered at 1180 ºC, as shown in Fig. 4 d).



a) b)

c) d)

Fig. 4 – Alloy/SiC interface morphology obtained for different sintering temperatures: 900ºC a) 1000ºC b), 1100 ºC c) and new phases formation from the SiC particle dissolution for sintering at 1180 ºC d).

The created alloy/SiC interface, at 1000 and 1100 ºC, is constituted by two zones: an internal zone near the SiC particles constituted by the FeSi + C phases and an outer zone of Fe3Si phase (zones 1 and 2 on Fig. 4 b) and c)). The phases chemical compositions (metallic phases), analyzed in zones marked on Fig. 4b) and c), are presented in table 2.

Table 2 – Phases chemical composition (in at.%) obtained in different zones (defined on Fig. 5) of the steel alloy/ SiC interfaces and at different sintering temperatures.

	1100 ºC		1000 ºC	
	Z1	Z2	Z2	Z2
Si	40,9	27,4	24,3	37,9
Cr	16,3	5,3	8,5	22,6
Fe	42,8	67,3	67,2	39,5

The reactions at the metal/SiC interface can be explained by the following sequence:

Inner zone

$$3Fe + SiC = Fe3(Si,Cr) + C \quad (1)$$

Outer zone

$$Fe3(Si,Cr) + Si = 2Fe(Si,Cr) + Fe \quad (2)$$
$$(Fe,Cr) + Si = Fe(Si,Cr) \quad (3)$$

Similar results were obtained by Pelleg (1999) and Tang et al. (2002) for the system Fe/SiC, at different temperatures. In this study the phases formed at the interface have significant amounts of Cr from the base alloy. However the Cr content changes along the interface (see results for two analyses on zone Z2 presented on table 2, for 1000ºC) indicating a dissolution process controlled by element diffusion through the interface.

Table 3 - Shear strength at different compacting pressure and sintering temperature.

Load (MPa)	Sintering temperature (ºC)	Shear strength (MPa)
	900	65
374	1000	50
	1100	139
	900	38
749	1000	84
	1100	215
	900	97
1123	1000	96
	1100	225

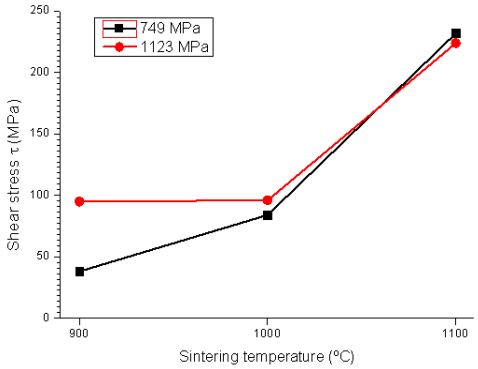


Fig 5 – Shear strenght obtaines with different tested compaction pressure and sintering temperatures

The effect of the compacting pressure and sintering temperature on the shear strength was studied. The results are presented on table 3 and Fig. 5. It is possible to verify that with the compression load of 374 MPa, the shear strength varies randomly. Thus, in this compaction pressure, the internal defects (namely the porosity) are more important than the process variables, compaction pressure and sintering temperature. This indicates that the used compacting pressure is not enough for the powder consolidation and that this effect is not compensated with the increase on the sintering temperature. Increasing the compaction pressure the results become more stable and changes linearly with the sintering temperature.

For the compacting pressure of 749 and 1123 MPa there is an increase of the shear strength with the sintering temperature. For sintering temperatures above 1000 ºC the effect of the compacting pressure is less important.

4. CONCLUSIONS

The alloy density, before sintering, is highly dependent on compaction pressure. An increase of the compacting pressure, from 300 to 700 MPa, results in a significant reduction on the internal defects. Further increases to 1123 MPa allows an alloy improvement, however, the changes are less significant.

The reinforcement particles react with the matrix alloy. The reaction is highly temperature dependent and for sintering temperatures of 1180 ºC complete particle (SiC) dissolution was verified. The reaction interface is constituted by two zones with increasing carbon contents from the particle to the matrix side. These two zones were identified as: inner zone is a

two phase zone with Fe3(Si,Cr) + C; outer zone is mainly of a Fe(Si,Cr) phase.

The mechanical properties are also dependent on compaction pressure and sintering temperature. Shear strength and rupture strength increases with the compaction pressure and the sintering temperature. There is a significant increase in those properties when compaction pressure changes from 374 to 704 MPa. A less significant increase was obtained by the change to 1123 MPa. A similar effect was obtained with the sintering temperature. The higher improvements were obtained with the change from 1000 to 1100 ºC.

5. REFERENCES

Yu, X.X. and Lee, W.B. 2000. The design and fabrication of an alumina reinforced aluminum composite material, Composites: Part A 31, p. 245–258.

Amaral Fortes, M. and Ferreira, P.J. 2003. Materiais 2000, Press, p.203.

Abenojar, J., Velasco, F., Torralba , J.M., Bas, J.A., Calero, J.A. and Marce, R. 2002. Reinforcing 316L stainless steel with intermetallic and carbide Particles, Materials Science and Engineering A335, p. 1–5

Pelleg, J. 1999. Reactions in the matrix and interface of the Fe–SiC metal matrix composite system, Materials Science and Engineering A269, p. 225–241.

Tang, W.M., Zheng, Z.X., Ding, H.F. and Jin, Z.H. 2002. A study of the solid state reaction between silicon carbide and iron, Materials chemistry and physics 74, p. 258-264.