SINTERING INTERMETALLICS COMPOUNDS MoSi₂ BY CO₂ LASER RADIATION

G. de Vasconcelos^{1*}; N.A.S. Rodrigues¹; R.C. Maia²

¹Instituto de Estudos Avançados do Centro Técnico Aeroespacial, São José dos Campos, SP ²Instituto Tecnológico de Aeronáutica – ITA, São José dos Campos, SP

Received: July 21, 2005; Revised: April 29, 2006

Keywords: laser sintering; green compacts; laser processing.

ABSTRACT

In this communication we present our results of the feasibility of sintering greens compacts of metallic powder of MoSi₂ by a CO_2 laser beam as the heating source. The main advantage of this technique is to promote a dense material in a reduced time when compared to the conventional sintering process. In order to sintering the MoSi₂ powder, greens compacts of 6mm of diameter and 1.6mm thickness were produced in a stell die in a uniaxial press at 100MPa and after, isostatic pressed at 350MPa. The micrograph of the samples exposed to the laser radiation performed by scanning electron microcopy (SEM) reveal the effciency of the sintering process and the X-ray diffraction of the powders confirmed the presence of single phase after and before laser processing. The average microhardness of these compacts reached near to 700 $Hv_{0.2}$ in the cross section to the laser irradiation, indicating the all sintering of the green copmpact.

1. INTRODUTION

The low density intermetallic compound MoSi₂ is currently being developed as a material for high temperature structural applications. This material exhibit excellent oxidation resistance at low and intermediate temperature, high melting point (2303K) and thermodynamic stability with a number of important ceramics materials. At the present, the methods used for making this material, are based on powder metallurgical process where powder of elements Mo and Si, are mixed and sintering in temperatures above of 1773K, and often requires hot isotatic pressing in order to achieve full densities [1].

In this communication we present our results of the feasibility of sintering greens compacts of metallics powders of $MoSi_2$ by a CO_2 laser beam as the heating source. The main advantage of this technique is to promote a dense material in a reduced time when compared to the conventional sintering process. In the laser sintering technique, the laser radiation promote a increase in the temperature distribuition of the grain, where the surface is at a much higher temperature. After some microseconds, temperature homogenisation takes place, and the whole grain is at a much lower average temperature [1]. The laser energy is tuned sufficiently to in-

duce a temperature raising in the powder as closed to the melting point of the metal particles but without exceeding it, in order to avoid melting, so that binding at the interfacial grain contact area occur. In this case, the principle of the sintering process with the metal powder is based on particle surface fusion at the temperature below the melting pointing. During the sintering process a neck forms between the adjacent powder particles. This reduces the surface area and increases the tendency of the powder aggregate [2]. The driving force for this is the reduction of the surface energy of the particles, and the densification is proportional to this reduction. But for higher temperatures or complete melting, one find that, owing to higher viscosity and surface tension effects, and the moltem metal tends to form a spherical, balltype structure wich has bigger dimensions than the particle size [2]. As the laser beam is usually bigger than the particle size, many particle get melted together and form a bigger spherical droplet, it is connected with other droplets only at certain points on its contourn [2]. Therefore the possibility is that porosity exists in the sintered structure. This process is workable only at slow speed but becomes difficult to realize in pratice, and post processing is required.

In order to eliminate post processing, the two-component method is used. This method is based in the mixing of the two powders one structural and the other a binder. The first one has a higher melting point than the other. The laser bean energy should be enough to raise the powder temperature between the melting point of the two metals, melting the binder and flow by driving forces through the pores between the solids particles. For better densification with reduced pores, the grain size of the binding particles should be smaller than that of the stuctural material. Nevertheless, the particles of smaller size lose their shape completely and form a cluster estructure. The large one are thus wetted by the smallers [3]. If the grains sizes of the bindings particles are substantially larger than those structural material particles, their partial melting may leave large pores, wich may not be filled by the rearrangement. This could also happen due to low laser energy, wich may result in residual porosity. On the other hand, if the laser energy is high, excess liquid formation may produce compact distortion [4]. An optimum laser energy and a appropriate binding material are essencial for a good sintered structure.

getulio@ieav.cta.br

2. EXPERIMENTAL PROCEDURE

The laser sintering experiments were performed with a CO_2 laser power range from 10 to 50W and the working beam speed varied from 20 to 900mm/s. The laser beam radiated the working surface sample from 1200 to 2000 turns as presented in Table 1. The laser beam focused by a lens of focal length of 200mm, produce a spot size of 300µm, by the side, it is desfocused to produce a beam of 2.5mm of diameter in the action point on the sample. This study was carried out in two steps, both with poligonals powders with a gaussian particle size distribuition of 1 to 50µm. It was used a mix of Mo + Si and $MoSi_2$ powder. In the first one, the Si acts as a binder. In two cases, a green compacts of 3mm diameter and 1.6mm of thickness were produced in a stell die in a uniaxial press at 100MPa and after, isostatic pressed at 350MPa. These compacted samples were exposed to the CO₂ laser radiation with differents intensity and times exposure (Table 1).

Table 1 - Experimental parameters and mean microhardness attained.

Laser	Time	Mean micro-	Heating	Process
intensity	exposure	hardness	pass	speed
(W/cm^2)	(min.)	(Hv _{0.2)}		(mm/s)
765	14	531 ± 53	1200	90
765	23	573 ± 32	2000	90
918	23	652 ± 65	2000	90
918	40	694 ± 50	2000	45

The surface temperature of the sample is controled by an optical pyrometer by adjusting of the velocity of the incident laser beam. The thermal gradient along the Z axis in the sample is evaluated by using a micro thermocouple inserted in the geometrical center of the transversal section of the sample. These medium values of the temperatures are presented in the Figure 1.



Figure 1 - Medium values of temperature on the surface sample in function of the laser beam velocity for two values of laser beam power. Measured by an optical pyrometer and by a micro thermocouple inserted in the middle of the transversal section of the sample.

A diagram for the laser beam processing is presented in the Figure 2. The beam overlap is about 10% of the laser beam diameter. After one pass exposure, the beam return to the initial position and repeat the heating operation.

After the interaction with the laser beam, the transversal section and the irradiated surface of the specimens were mechanically polished down to 1 μ m. The characterization of the treated zones was performed by a scanning electron microscopy (SEM), optical microscopy (OM), microhardness and X-ray analysis.



a) diagram to the laser sintering process. b) Heated Affected Zone -HAZ by the laser beam action.

Figure 2 - A diagram for the laser sintering process.

3. EXPERIMENTAL RESULTS

SEM of used powder are presented in the Figure 3, and reveal a poligonal shape powder. The XRD patterns of the started MoSi₂ powder are shown in the Figure 4, the major phase is MoSi₂, before and after the laser processing. A short peak of a silicides were observed near to 23 DEG in the laser processed sample as shown in the Figure 4.



Figure 3 - SEM of the pressed MoSi₂ powder with a pre heating, to reveal the grain shape. A poligonal powder can be seen.

A microstructure of the cross section of the heated afected zone of a sample of $MoSi_2$ powder after laser processing is presented in the Figure 5. A laser beam intensity were 1020W/cm² with a diameter of 2.5mm and time exposure

23min. and laser beam velocity of 45mm/s were used. No sample surface fusion were observed by optical microscopy.



Figure 4 - XRD patterns of MoSi₂ powder before and after laser sintering .



Figure 5 - Heated afected zone microstructure by the laser processing. Time exposure 23min., laser intensity 1020W/cm². The temperature on the surface sample reached 1800K ±30K.

4. CONCLUSIONS

It has been shown in this study that the laser beam of low energy can be an attractive method to sintering intermetallic powders of MoSi₂. The differences in the sample surface temperature measured by optical pyrometer and thermocouple are nearest to 5% and do not affected the sample densification. The final product obtained, for 1020W/cm² is hard, with a solid structure with reduzed porosity, and was obtained in a few minutes of laser radiation (time exposure 23 min). The efficiency of the laser sintering process can be proved by the necks formations presented in the Figure 5. The experiments related to Mo+Si powder were rejected, because a liquid phase takes place when the temperature on the surface sample overcomes the silicon temperature melting point, although the MoSi₂ phase were confirmed by XRD analysis.

REFERENCES

- HIDOUCI, A.; PELLETIER, J.M., Materials Science and Engeneering A 252 (1998) 17.
- KATTHURIA, Y.P., Surface and Coating Technology 116-119 (1999) 643.
- IGNAT, S.; SALLAMAND, P.; NICHICI, A.; VANNES, B.; GREVEY, D.; CICALÃ, E., Intermetallics 11 (2003) 931.
- 4. FISCHER, P.; ROMANO, V.; WEBER, H.P.; KOLOSSO, S., *Thin Solids Films* 453 (2004) 139.