

DENSIFICATION AND MICROSTRUCTURE FORMATION OF SYSTEMS BASED ON HSS M2-SiC-Cu AT LOW TEMPERATURE VACUUM SINTERING

A. Simchi, M. Khakbiz

Abstract

The densification and microstructural features of M2 grade high speed steel powder compacts with SiC and Cu additions were studied. The test specimens were prepared using uniaxial cold compaction followed by vacuum sintering at relatively low temperature, i.e. 1100-1120°C. Microstructural analysis of sintered samples and identification of phases were made using optical microscopy, SEM and EDX-analysis. The density and the mechanical specifications of the specimens were evaluated in accordance to the standard methods. The results show that the compressibility of the steel powder enhances with addition of tough Cu powders, while introducing the SiC hard particles has detrimental influence on the consolidation. In contrast, it was found that during vacuum sintering at low temperature the influence of Cu on the densification is marginal; instead, SiC addition enhances the sintering kinetics considerably. Based on the results of metallographic study, this behavior is attributed to the reaction between M2 matrix and SiC particles at around 1100 °C, resulting in the formation of low melting Fe-Si-C phase. Although high densification rate was obtained for the specimens containing SiC particles, grains- and carbides- coarsening were likely to occur. By varying the composition of the mixture and sintering condition, the structure, density, and properties such as wear resistance, resistance to hot crating and hot hardness may be optimized for a given application.

Keywords: *high speed steel, SiC, compaction, sintering, microstructure*

INTRODUCTION

High speed steels (HSS) are used for applications that require strength and hardness combined with the ability to withstand high temperatures, e.g. cutting tools, and for demanding cold work applications such as fine blanking tools and dies [1]. Although these materials have been produced by conventional steel making methods for several years, the powder metallurgy route has contributed to dramatic cost reductions in mass production of HSS [2]. In fact, during the last couple of decades the process has been satisfactorily developed, for producing parts with improved properties and lower production costs than the conventional methods. For instance, manufacturing of wear resistant automotive and aerospace components such as valve seat inserts for internal engines and bearing materials for aerogas turbines has been done successfully [3-5]. The main advantage of using this technique refers to the ability of obtaining a fine and uniform microstructure in which segregation is reduced to insignificant levels [2,6]. Accordingly,

better strength and grinding characteristics as well as higher hardness and resistance to wear can be achieved.

Nowadays, cold compaction and vacuum sintering of prealloyed HSS powders to full density is a well established process [3]. The powders are pressed to a density of about 70 % theoretical, and are sintered in the temperature range 1240-1330°C to promote the supersolidus liquid phase sintering process. In spite of the advantages of achieving full density, it is well known that application of the supersolidus sintering process of HSS requires keeping a close interval of temperature of liquid phase formation [7]. This problem suggests an important field of study of densification by formation of a secondary liquid phase using several additives. For instance, introduction of copper phosphide is successful in lowering the sintering temperature and fully dense parts can be produced using continuous mesh belt furnaces [8-10]. The vacuum sintering of systems based on HSS M2 powder chemically coated with Cu and graphite additions was successfully implemented [11, 12]. The positive influence of FeB powder on the densification of HSS was also studied [10].

It is known that carbon and silicon additions to tool steels significantly affect the sintering temperature by lowering the solidus temperature. However, blending of more than 0.15 to 0.2 wt% C may cause several detrimental effects, such as nonuniform distribution of carbon, variable response to sintering, variable part-to part density, and part distortion [6]. On the other hand, admixed silicon does not homogenize rapidly because of its low diffusivity; consequently, it usually causes incipient melting. One possible way is to use SiC particles as sintering aid instead of elemental graphite and silicon powders. This work offers better blend homogeneity during mixing, which in fact may suppress the detrimental influences of using elemental powders. The aim of this article is to report on the interaction that occurs during processing of M2 grade high speed steel with SiC particles. The influence of Cu addition was also studied.

EXPERIMENTAL PROCEDURE

The starting powders used for manufacturing the sintered materials were water atomized M2 grade HSS powder from Hoganas, Belgium; SiC powders with mean powder particle size of 14, 18 and 24 μ m from Zamin Tavana Co., Iran; water atomized copper powder from MPM Co., Iran. The compositions, and some characteristics of HSS powder used, were summarized in Table 1.

Tab.1. Composition and properties of M2 grade high speed steel examined

Composition [wt%]	C	Cr	W	Mo	V	Mn	Si	O+N
	0.83	4.19	6.13	4.08	1.925	0.27	0.19	*
Powder sieve analysis [wt%]	<45 μ m		45 - 53 μ m		53 - 75 μ m		75 - 106 μ m	
	35.6		11.9		21.8		17.8	
Properties	Apparent density g cm ⁻³		2.55		Hall flow s/ 50g		29.7	

The composition of the investigated powder mixtures was 1-5 wt% SiC and the balance of water atomized based powder. The composite powders were blended in a Tumbling mixer for 30 min. A small batch of the HSS powder was also modified by coating particles with copper by a cementation reaction in an aqueous CuSO₄ solution as cited in [8]. In this process, the steel powder was poured into stirred CuSO₄ solution at ambient temperature until the blue color of the solution had completely disappeared, and then filtered off, rinsed with water and acetone, and dried at 70°C. The copper content was

adjusted to 2 wt% by controlling the powder mass. Moreover, some runs were made by 2 wt% elemental Cu additions.

The powder blends were uniaxially cold compacted to cylindrical (25 mm diameter and 6 mm high) and rectangular (90x10x10 mm) specimens at 550 MPa, die-wall lubrication being afforded. The compacts were then sintered in a laboratory tube furnace in a vacuum of the order of 10^{-2} torr at 1120°C for 20 and 60 min. It is pertinent to point out that an attempt to process the green compacts at higher temperatures resulted in compact slumping as well as melt oozing out of the specimens containing high amounts of SiC particles. After sintering, the samples were furnace cooled to ambient temperature. The mean cooling rate was around 10 K/min in a range from 800 to 580°C.

The density of the sintered samples was measured by the water displacement method. A surface impregnation technique with a water stop spray was employed to prevent penetration of water into the pores open to the specimen surface. The hardness of the samples was evaluated by using the Vickers method under 0.05 and 30 kg force. Microstructural analysis of sintered microstructure and identification of phases were made using optical microscopy, SEM and EDX-analysis.

RESULTS AND DISCUSSION

Density

The green density of the investigated material systems is shown Fig.1. One can see that the copper coating decreases the green density. However, addition of elemental copper powder has a positive influence on the densification. The advantage of blending hard high speed steel particles with tough and highly compressible powders to enhance the compressibility has been already established [5]. On the other hand, Strobl and Danninger [8] have shown that Cu-coated M2 powders were more difficult to compact in comparison to the base powders, and the green bodies being comparatively fragile. Similar results were found in this study. It may be due to surface condition of the coated powder, which influences friction, interlocking, and cold welding between particles.

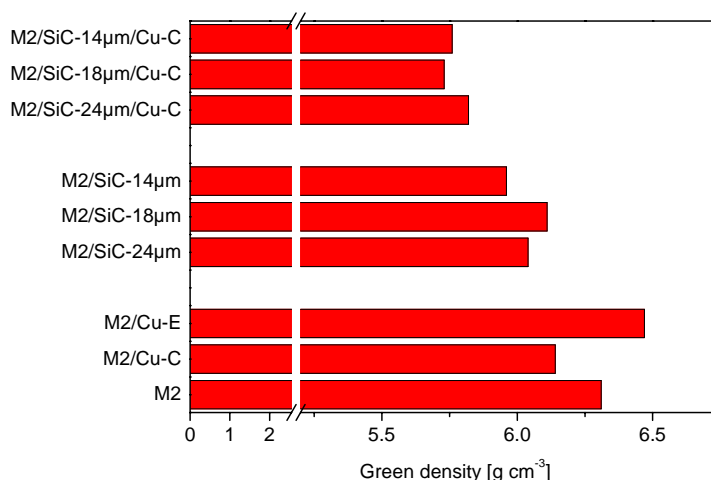


Fig.1. The green density of the investigated material systems compacted at 550 MPa; Cu-C: copper coated; Cu-E: elementally prepared by admixing with copper powder.

From Figure 1, it is also visible that additions of SiC particles to the M2 base powder adversely affect the compressibility. When the M2/SiC composite is accompanied by Cu coating, the green density is reduced further. It seems that the adverse effect of adding SiC and Cu coating was superimposed. It is evident that the SiC particles are rigid bodies that do not deform to fill in the voids. On the other hand, the M2 particles are comparatively more ductile plastic bodies. It is therefore expected that additions of SiC particles suppress the compressibility of the steel powder. It is obvious that this effect depends on the size and the amount of the hard particles added. Figure 2 depicts the influence of SiC addition on the green density of M2/SiC composite compacts. Here, the relative density was used to omit the effect of the lower density of the carbide particles. On can notice that the adverse effect of SiC addition is more pronounced for the composite compacts containing >2 wt% SiC. On the base of the percolation theory [13,14], which analyzes the connectivity of a two-body system, it can be noticed that the 2 wt% SiC should be the percolation threshold. It means that at higher SiC additions, the ceramic particles are not distributed predominantly as isolated grains spanning M2 particles, i.e. larger SiC clusters are formed. This leads to lower green density and reduces the green strength.

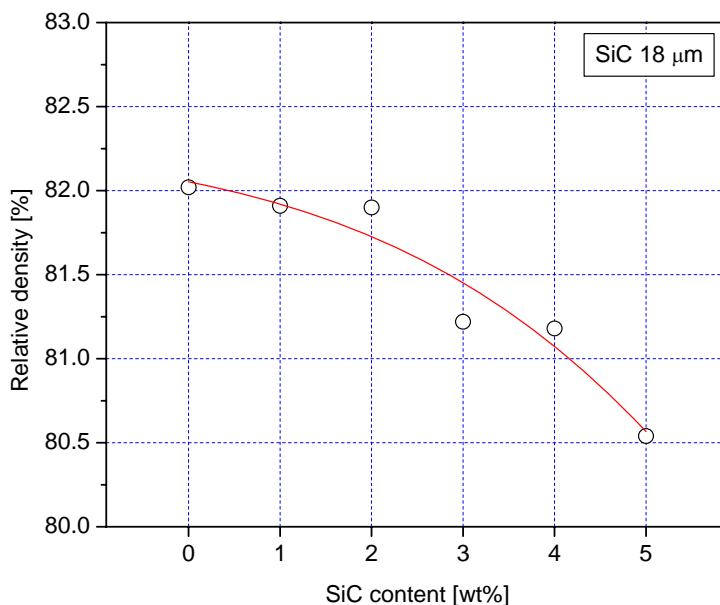


Fig.2. Effect of SiC addition on the green density of M2 grade HSS in relative to pore free M2/SiC powder compacts.

The effect of sintering on the density of the investigated material systems is given in Table 2. It is known that the optimum vacuum sintering temperature of cold compacted M2 grade HSS was established as 1243-1255°C, but also around 1330°C [15]. For ultra-fine M2 powders full density could be achieved at approximately 1200°C [10]. Therefore, sintering at a temperature as low as 1120°C does not impact the density of M2 base powder. On the other hand, the Cu content does not markedly affect the optimum sintering temperature which is well comparable to that of the plain HSS used as basic powder.

Tab.2. Effect of sintering (1120°C, vacuum, time: 20 and 60 min) on the density [gcm^{-3}] of the investigated material systems compacted at 550 MPa.

Material	Green compacts	Sintered for 20 min	Sintered for 60 min
M2	6.31	6.34	6.44
M2/Cu (elemental)	6.47	6.68	6.76
M2/Cu (coated)	6.14	6.24	6.83
M2/SiC (24 μm)	6.04	7.26	7.61
M2/SiC (18 μm)	6.11	7.59	7.20
M2/SiC (14 μm)	5.96	6.9	6.58
M2/SiC (24 μm)/Cu	5.82	7.52	7.12
M2/SiC (18 μm)/Cu	5.73	7.07	7.68
M2/SiC (18 μm)/Cu	5.76	6.85	7.06

However, the results show that additions of SiC particles strongly influence the densification of M2 base compacts. The effect also markedly depends on the particle size of the ceramic particles as well as sintering time (Fig.3). It is well known that adding inert, rigid inclusions to a powder system retards sintering [16,17]. The inert particles not only reduce the volume of densifying material, they also create a hydrostatic tensile stress in the matrix which opposes the sintering stress. This causes de-sintering and crack-like void formation [18]. In the case of HSS/TiC/TiN composites Bolton and Oliveira [19] have found that additions of ceramic particles reduce the sintering rate. They have also shown the sintering rate is greater for the composites containing coarser TiC particles at low temperatures of $T_s < 1160^\circ\text{C}$. However, good adhesion at the ceramic/matrix interface was produced by the formation of an interfacial layer of vanadium rich carbide due to wetting by the liquid phase. This results in good sintering behavior and mechanical strength.

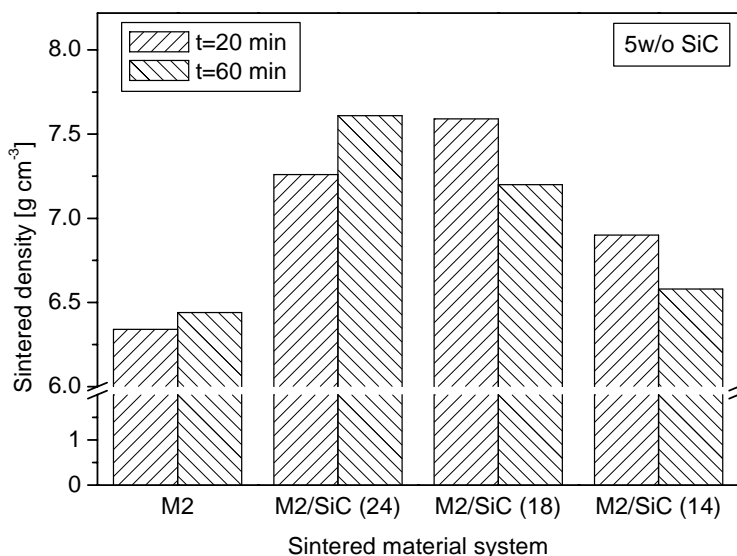


Fig.3. Effect of SiC particle size and sintering time on the density of M2 compacts sintered at 1120°C in vacuum; the number in parenthesis indicates the mean particle size of SiC.

Given the observations of the present work, one can understand that during the processing of the composites, the M2 matrix interacted with the SiC particles resulting in the formation of a low melting phase. It noteworthy to mention that the sintering behavior of HSS is strongly influenced by variations in carbon content due to the effect on the solidus temperature [6]. Therefore, it seems reasonable to suggest that such a big difference in sintering behavior of the investigated materials is due to 1) the formation of austenite/SiC melt upon sintering or 2) complete or partial dissolution of SiC particles into the matrix. The latter results in a change in the dissolved carbon and silicon of the metal matrix, eventually leading to the formation of the liquid phase at a temperature as low as 1100°C. The austenite/silicon carbide eutectic is formed at the same temperature as well [20]. In any case, when the sintering of M2 compacts containing >4 wt% SiC was attempted at temperatures higher than 1120°C, the sintered specimens showed signs of molten metal oozing out of the compacts, and in some cases complete melting, due to an increased amount of the liquid being formed. The results are comparable to the outcome of the study carried out by Patankar and Tan [20] for a SiC/316 stainless steel composite. They have found that during processing of the composite, such a liquid phase will be formed around 1100°C, resulting in a high sintering rate. However, the use of a higher sintering temperature resulted in the complete melting of the compact.

Microstructure

The effect of the ceramic additions on the microstructural development of M2/SiC compacts is shown in Fig.4. It can be seen that the microstructure of the M2 compact used as basic powder consists of fine carbides uniformly distributed on the grain boundaries of the austenite matrix as well as small intragranular carbides (Fig.4a). The size of the grains is in the order of 10 μm . The prior particle boundaries (PPBs) are also visible, which determine insufficient sintering. In contrast, the microstructure of the steel containing SiC particles consists of relatively large grains (about 20-30 μm) covered by a white phase. The carbide phases are also coarser in comparison to M2 base compact. However, it was found that more/larger pores exist in the microstructure of the composites containing 14 μm SiC particles.

Figure 5 shows the SEM micrograph of the microstructure taken from the back-scattered electrons. The microstructure of the base material consists of angular M_6C and round MC carbides in a matrix, which was predominantly austenite (due to the relatively slow cooling rate in the sintering furnace). The PPBs are also visible. The microstructure of M2 compacts containing SiC particles consists of relatively coarser grains, an eutectic phase at the grain boundaries, distributed intragranular round carbides, and coarse angular carbides at the grain boundary near the eutectic phase (Figs.5b and 5c). In Figure 6, the results of EDX analysis of the phases for the M2/SiC-24 μm compacts are shown. The eutectic phase seems to be predominantly a Fe-C-Si alloy containing Mo and Cr. The angular phase is complex M_6C carbide (typically Fe-W-Mo-C) and the round phase is MC carbide (typically V-W-Mo-C). Similar results were obtained for the compacts with finer SiC particles. However, the outcomes of density measurement (Fig.3) determine that an addition of 14 μm SiC particles has comparatively less influence on the densification. It seems that the amount of liquid being formed is lower compared to the other compacts containing coarser SiC particles (Fig.4). It may be due to agglomeration of the fine SiC particles during processing, which influences the overall surface of the metal/ceramic interface. On the other hand, the surface of the finer SiC particles is more likely to be contaminated, e.g. by surface oxidation during SiC processing.

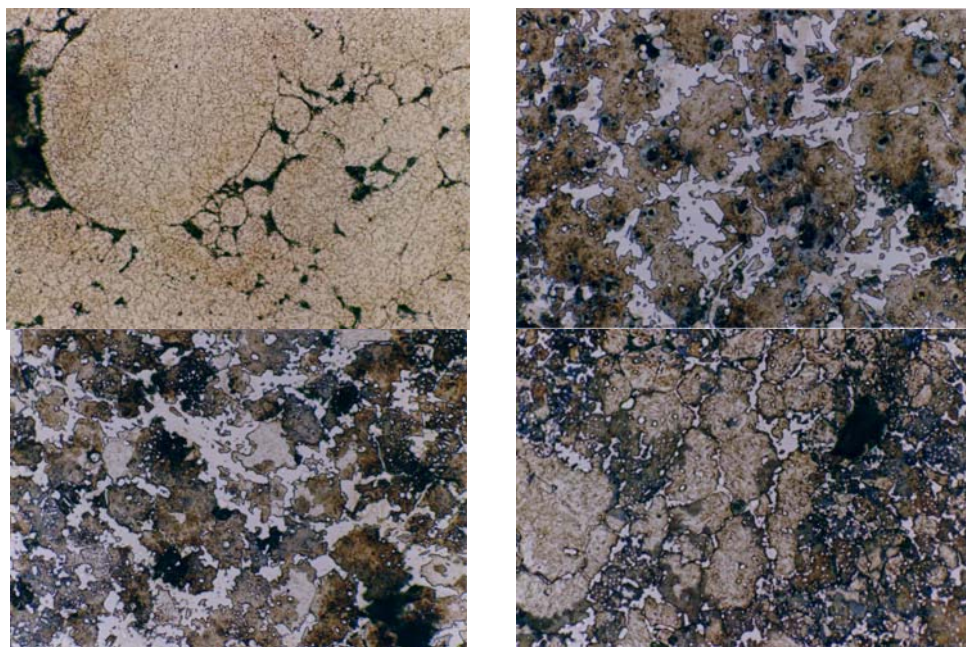


Fig.4. Metallographic sections of the investigated materials compacted at 550 MPa and sintered at 1120°C for 20 min in vacuum.

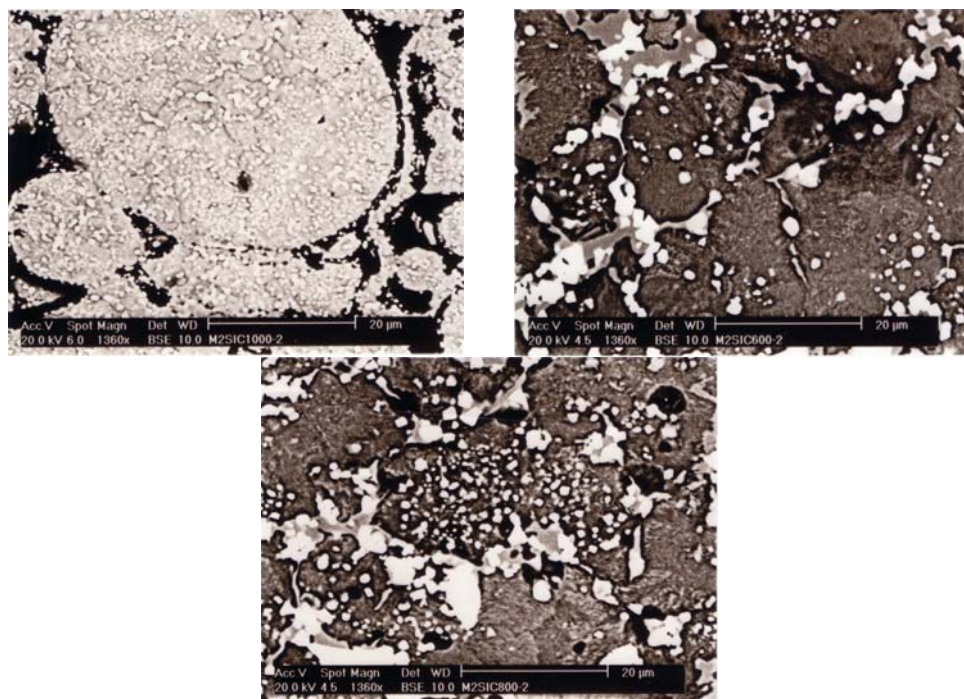


Fig.5. Back-scattered electron micrograph of the investigated materials compacted at 550 MPa and sintered at 1120°C for 20 min in vacuum.

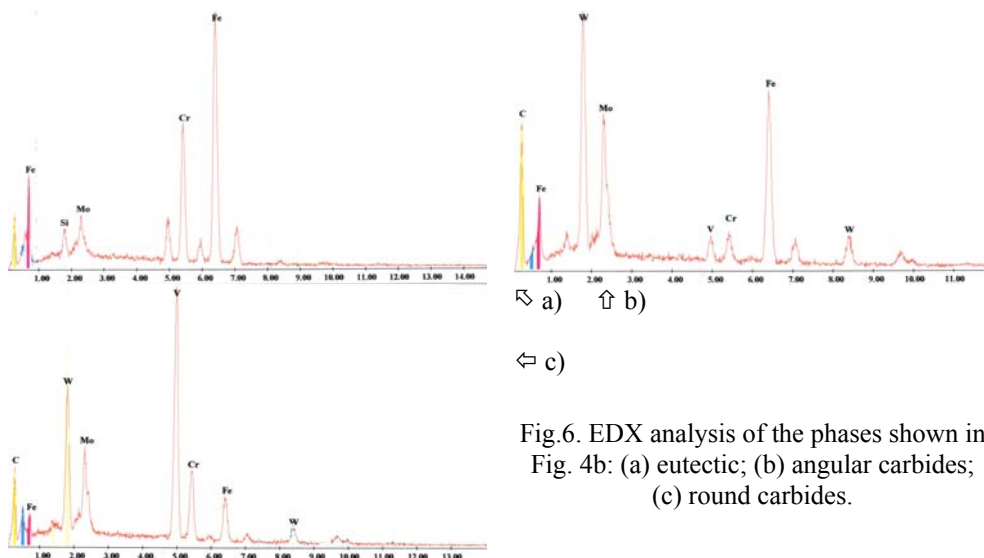


Fig.6. EDX analysis of the phases shown in Fig. 4b: (a) eutectic; (b) angular carbides; (c) round carbides.

Mechanical properties

Figure 7 depicts the mechanical properties of the investigated materials as a function of SiC content. Here, it should be stated that the microhardness test was performed on the metal matrix. These results clearly show that additions of SiC particles lead to better mechanical properties. Of interest, both hardness and the bending strength were improved. This is obviously due to higher sintered density and a harder metal matrix.

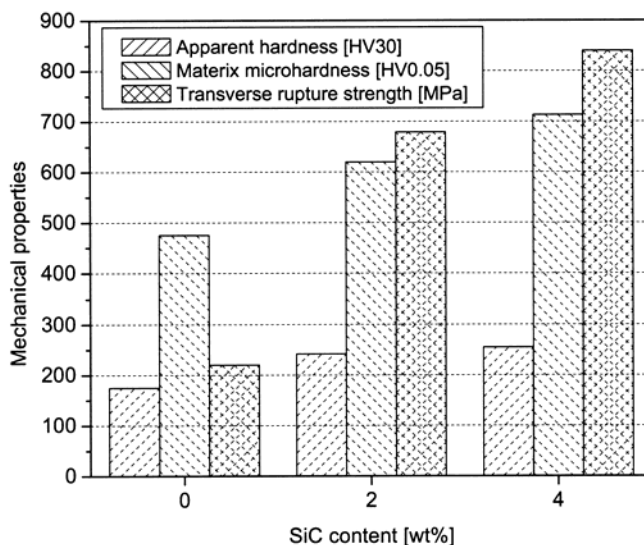


Fig.7. Effect of SiC (18 μ m) additions on the mechanical properties of sintered M2 grade HSS compacts (1115°C, 20 min, vacuum).

Tab.3. Hardness values of the sintered HSS M2 base material and of the SiC containing compacts (1120°C for 20 min in vacuum).

Material	Density [g cm ⁻³]	HV0.05	HV30
M2	6.34	730 ± 31	263 ± 20
M2/SiC (24 μm)	7.26	770 ± 37	447 ± 38
M2/SiC (18 μm)	7.59	655 ± 22	511 ± 31
M2/SiC (14 μm)	6.9	967 ± 30	427 ± 20

The effect of SiC particle size on the hardness values of the sintered M2/SiC compacts are given in Table 3. The micro-hardness was taken from the metal matrix of the composite. The hardness values for plain M2 grade HSS are also included for comparison. One can notice that the macro-hardness of the steel compact containing 18 μm SiC is higher than those of the others. Again, when considering the sintered density it seems reasonable to suggest that this big difference in the hardness values is caused by the density effect. The effect of the metal matrix hardening, as detected by the micro-hardness test, should be also taken into account.

However, microstructuring of the investigated materials demonstrated that carbide coarsening during the sintering cycle is likely to occur. If the M2 matrix interacts with SiC particles and carbon dissolves into the iron lattice, then it would be expected that more austenite be retained during slow furnace cooling. Surely, these phenomena adversely affect the strength and hardness of the material. On the other hand, it is quite normal to employ the quench-tempering heat treatment on high speed steels in order to improve the mechanical properties. Therefore, the measured mechanical properties do not give the full potential of the investigated materials. More research work is underway to optimize the sintering process and the resulting mechanical properties, which is not considered in the present work.

CONCLUSIONS

The effects of SiC and Cu additions on the sintering behavior and the resulting microstructural features of M2 grade high speed steel powder were studied. It was found that adding hard ceramic particles suppresses the compressibility of the base steel powder. This effect is more pronounced for the compacts containing >2 wt% SiC. This is the threshold value in which at higher SiC amounts, the SiC particles are not distributed predominantly as isolated grains spanning M2 particles, i.e. larger SiC clusters are formed. This leads to lower green density and reduces the green strength. On the other hand, the results showed that the copper coating decreases the green density, while additions of elemental copper powder has a positive influence on the densification.

Furthermore, it was found that during sintering the M2 matrix interacts with the SiC particles. This leads to the formation of eutectic Fe-C-Si melt which enhance the sintering rate. The mechanical properties, e.g. hardness and transverse rupture strength, are thus improved. This is obviously due to higher sintered density and harder metal matrix. Here, it is pertinent to point out that the particle size of the ceramic particles strongly influences the densification behavior. On the basis of the experimental observations, the materials containing 18 μm SiC particles were found to show more reasonable results with regard to the densification and thus the mechanical properties measured.

Microstructural analyses were also shown, that upon sintering grain growth was likely to occur. Furthermore, carbide phase coarsening was also detected. Since the mechanical properties of tool steels strongly depend on the size and uniformity of the

carbide phases, care must be taken into account to optimize the microstructure of the compacts. By varying the composition of the mixture and sintering condition, the structure, density, and properties such as wear resistance, resistance to hot crating and hot hardness may be optimized for a given application.

Acknowledgement

The authors are grateful for the grant provided by the Vice-Chair Office for Research and Technology, Sharif University of Technology. They are also thankful to Prof. H. Danninger and Dr. S. Strobl, Vienna university of Technology, for their input during valuable discussion. The support of MPM Co., Mashad, Iran for providing the materials is also acknowledged.

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