JOURNAL OF MATERIALS SCIENCE **33** (1998) 3219–3225

Wear and thermal behaviour of M2 high-speed steel reinforced with NbC composite

W. C. ZAPATA, C. E. DA COSTA
Departamento de Engenharia Mecanica, Universidade do Estado de Santa Catarina, Brazil
J. M. TORRALBA
Departamento de Ingeniería, Universidad Carlos III de Madrid, Leganés, Spain

This work studies a high speed steel composite reinforced with NbC and using Cu_3P as sintering activator. High speed steels can be used as cutting material and their cutting capability could be improved if hardness, and as a consequence, wear behaviour is increased. One way for increasing these properties is to reinforce them with hard particles. NbC have been demonstrate like one good option, but with the problem of its low interaction with the HSS matrix. One way for solving this low reactivity is to add a sintering activator that interacts with the matrix and increases the joint with the reinforcement. The thermal behaviour has been studied in order to optimize the sintering parameters, as well the best heat treatments conditions. This behavior has been determined through differential dilatometry. The wear behaviour has been studied through a pin on disk test using Al_2O_3 of high hardness as countermaterial. Finally, mechanical properties and complete metallographic study have been performed. © *1998 Kluwer Academic Publishers*

1. Introduction

In comparison with wrought high-speed steels (HSSs), powder metallurgical (P/M) HSSs have a more uniform microstructure with fine and well-distributed carbides, which prevents coarsening or a skeleton-type eutectic surrounding the ferrite grains. P/M HSSs have no segregation of alloying elements, at microstructural levels nor at structural levels. Microstructural problems in P/M HSSs could have their origin in an inadequate sintering temperature that promotes undesired melts and a subsequent eutectic network on cooling [1]. However, with an optimal sintering process, P/M HSSs will show better characteristics than the same grades of HSSs made by conventional methods, and some HSSs can only be obtained by P/M methods.

One way to improve the properties of P/M HSSs, mainly their wear behaviours, is to use ceramic reinforcements such as alumina or carbides [2–5]. Some workers have shown that these reinforcements improve not only the wear behaviour but also properties such as fracture toughness. In this way, HSSs are used as the matrix of a cermet which is reinforced with several kinds of particle. Thin grains [6] of well-dispersed alumina of small size (less than 10 μm) improves the wear strength of HSSs. In [7], some ceramic materials have been used in a coated state with an important improvement in sinterability and bonding between matrix and reinforcement. Another procedure [8–11] utilized is to dilute HSSs with low-iron alloys or Cu_3P to lower the sintering temperature to those of industrial belt furnaces for the manufacture of structural parts at conventional atmospheres (N_2–H_2 mixes). These new hybrid materials will lose some high-temperature resistance and hardness, but their properties are adequate for most wear applications at low production costs. Most of these types have been reviewed in [12]. Finally, another way to improve the cutting capability of P/M HSSs is to use coatings and surface treatments: TiN, TiC and Al_2O_3 [13]. TiN improves the life of a cutting tool between three and ten times, depending on the application, and allows coated HSSs to be used in some cemented carbides.

The present work examines a composite-base HSS reinforced with NbC, using Cu_3P as the sintering activator. The grade of HSS studied is M2, and its use came from the idea developed by Sintertech and manganese bronze [14, 15]. These patents associate the use of Cu_3P with M2 to reduce the sintering temperature by about 100 °C. With this additive, it is possible to sinter at below 1150 °C which is the usual limit for conventional belt furnaces. As already stated, this dilution reduces hardness and wear capability, but these are recovered through ceramic additives. Sintering occurs with a liquid phase whose relation with the ceramic reinforcement is essential to the interface quality and the composite properties. The relative wettability between both phases and the reaction between them must be evaluated and balanced. Jouanny-Tresy *et al.* [16] showed that the use of coated ceramic particles improves the wettability and in consequence the bond between the HSS and the reinforcement.

This work includes a dilatometric study, and the mechanical and wear behaviour of the materials in the sintered and heat-treated states.

0022–2461

2. Experimental procedure

An M2 HSS grade was used (from Coldstream, Belgium), together with niobium carbide (from CBMM, Brazil), Cu_3P and natural graphite. The characteristics of the different powders are as given in Table I.

From previous work [17] and from the literature [4, 7, 11] the amount of Cu_3P was selected and the following mixes (by weight) were made in a laboratory mixer: A, M2–7% Cu_3P; B, M2–7% Cu_3P–3.5% NbC; C, M2–7% Cu_3P–5.0% NbC; D, M2–7% Cu_3P–10.0% NbC. 0.2 wt% of natural graphite was added to all the mixes to prevent carbon losses. The mixed materials were die pressed (in a floating die) at 700 MPa using zinc stearate as the die lubricant. Materials were consolidated as transverse rupture strength bars according to ASTM Standard B-528. Green densities between 6.26 and 6.33 $g\,cm^{-3}$ were obtained in all cases.

Sintering was carried out in a laboratory furnace at 1150 °C for 60 min in a N_2–5% H_2 atmosphere. The heating rate was 15 °C min^{-1} up to 900 °C and 5 °C min^{-1} to the sintering temperature. Cooling was done slowly inside the furnace. Sintered densities of between 7.2 and 7.6 were obtained.

After sintering, heat treatments were carried out as follows: heating cycle, heating, at 5 °C min^{-1}, at 450 °C for 5 min, at 850 °C for 5 min, and up to the austenitizing temperature (950, 1000, 1100 and 1150 °C) for 10 min; quenching in oil (30 °C); tempering at 550 °C for 120 min, four times.

The dilatometric study was performed in a Bähr dilatometer under vacuum conditions to study the sintering behaviour of the materials and the best tempering parameters.

The sintered density and the mechanical properties (transverse rupture strength, hardness and elongation) were measured. Microhardness tests of all the materials were also made, as well as a wide microstructural study by conventional optical microscopy and scanning electron microscopy.

The wear tests were performed at room temperature (with relative humidity below 30%) using a Centre Suisse d'Electronique et de Microtechnique "pin-on-disc" apparatus. The disc was made of the material under study (in the sintered condition and in each heat treatment condition). As a counterpart, alumina with a Vickers hardness of 2500 HV was used (with a hemispherical extremity of 6 mm internal diameter). No lubricant was used. A normal load of 10 N and a constant sliding velocity of 0.1 $m\,s^{-1}$ (by adapting the rotary speed of the disc to the diameter of the track) was selected. During the test the tangential force was measured and continuously recorded as the friction coefficient. The specific wear rates were calculated for both pin and disc using the conventional expression

$$\text{specific wear rate } (m^2\,N^{-1}) = \frac{\text{material loss } (m^3)}{\text{normal load (N)} \times \text{sliding distance (m)}}$$

The material loss was calculated by measuring the wear facet diameter on the pin and from the weight loss of the disc.

TABLE I Characteristics of the various powders

	Chemical composition (%)							Sieving size (μm)
	C	W	Cr	V	Mo	P	Cu	
M2	0.88	6.44	4.10	1.72	4.96	—	—	10–150
NbC	13.2	—	—	—	—	—	—	<63
Cu_3P	—	—	—	—	—	14.7	Balance	<63

3. Results

Figs 1 and 2 show the method for determining the optimum austenitizing temperature. The mechanical properties (hardness and transverse rupture strength) are shown as functions of the austenitizing temperature.

Figs 3, 4 and 5 present the dilatometric study of all the compositions: Fig. 3 the sintering behaviour and Figs 4 and 5 the influence of further tempering of the materials after quenching.

Figs 6 and 7 show the influence of heat treatment on hardness and transverse rupture strength of the materials, using the optimized austenitizing temperature. Fig. 8 shows the matrix microhardness as a function of different heat treatments.

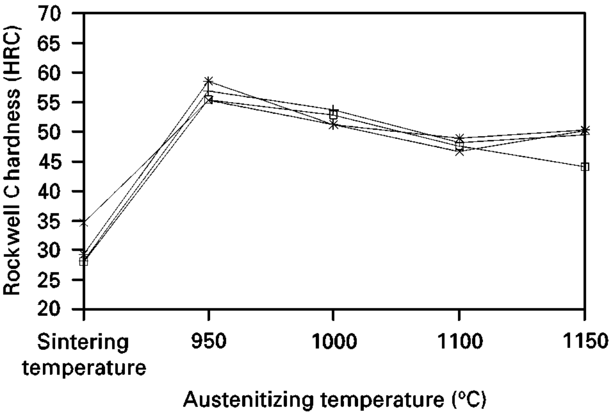

Figure 1 Hardness as a function of austenitizing temperature for all the materials studied. (—+—), M2; (—✱—), M2 + 3.5% NbC; (—⊟—), M2 + 5% NbC; (—×—), M2 + 10% NbC.

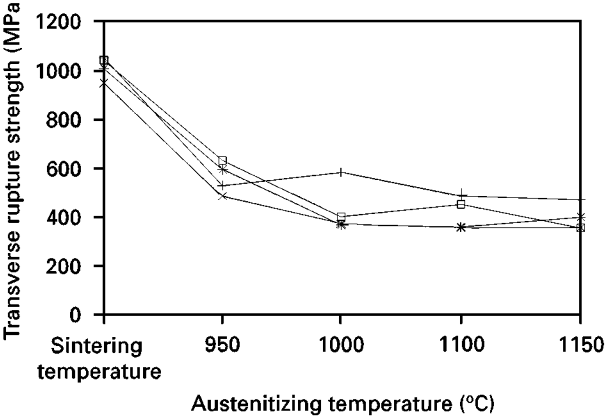

Figure 2 Transverse rupture strength as a function of austenitizing temperature for the materials studied. (—+—), M2; (—✱—), M2 + 3.5% NbC; (—⊟—), M2 + 5% NbC; (—×—), M2 + 10% NbC.

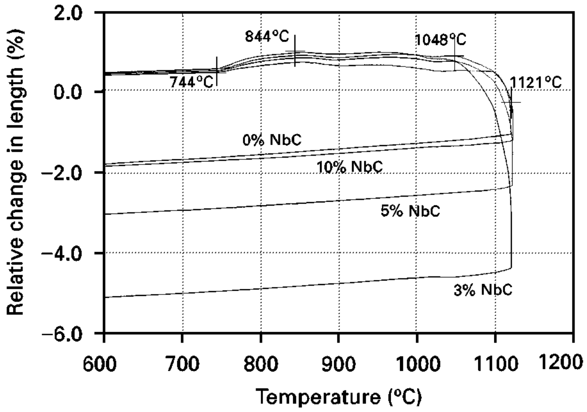

Figure 3 Dilatometric study of materials: vacuum sintering.

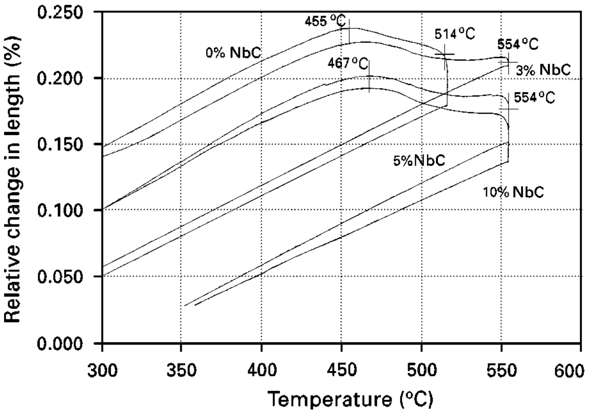

Figure 4 Dilatometric study of quenched materials: first tempering.

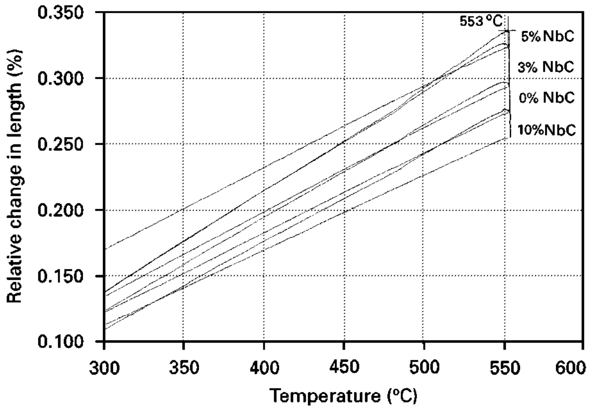

Figure 5 Dilatometric study of tempered materials: second tempering.

Figure 9 presents the wear behaviour of these materials and their relative mechanical properties.

Finally, Figs 10–14 show the microstructural features.

4. Discussion

The sintered densities were in some cases close to 80% of the theoretical density. Some reviewed studies report higher densities but using higher compacting pressures (850–1000 MPa); so the results must be analysed within the limitation of the respective densities. Fig. 1 shows how, in all cases, the best results are for

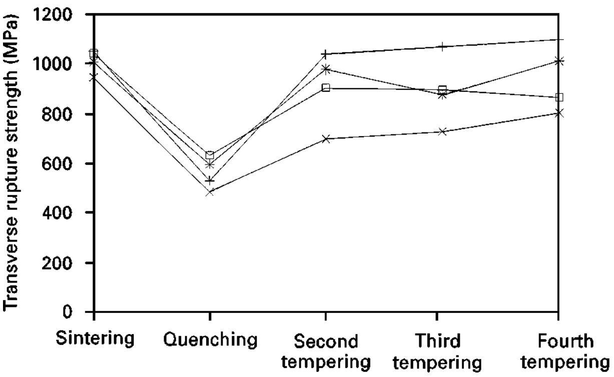

Figure 6 Hardness of materials under study at different heat treatments. (—+—), M2; (—∗—), M2 + 3.5% NbC; (—⊟—), M2 + 5% NbC; (—×—), M2 + 10% NbC.

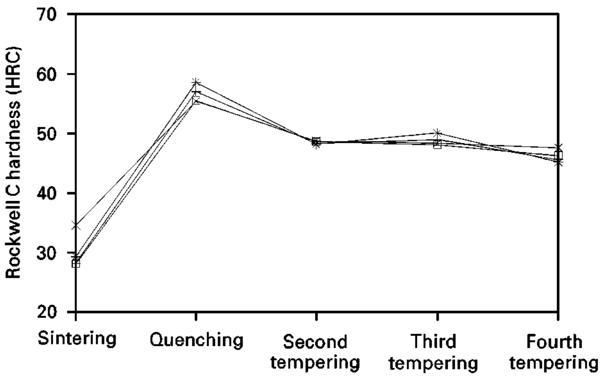

Figure 7 Transverse rupture strength at different heat treatments. (—+—), M2; (—∗—), M2 + 3.5% NbC; (—⊟—), M2 + 5% NbC; (—×—), M2 + 10% NbC.

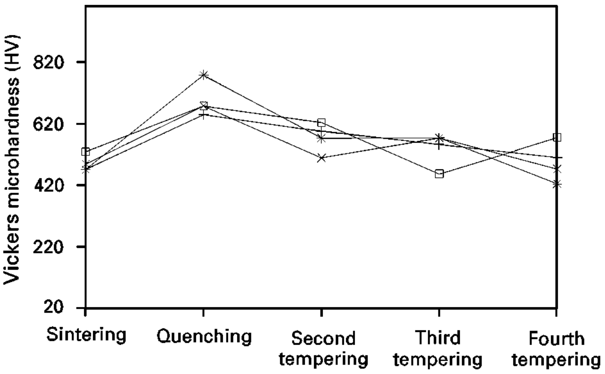

Figure 8 Microhardness of different microconstituents present in the materials studied. (—+—), M2; (—∗—), M2 + 3.5% NbC; (—⊟—), M2 + 5% NbC; (—×—), M2 + 10% NbC.

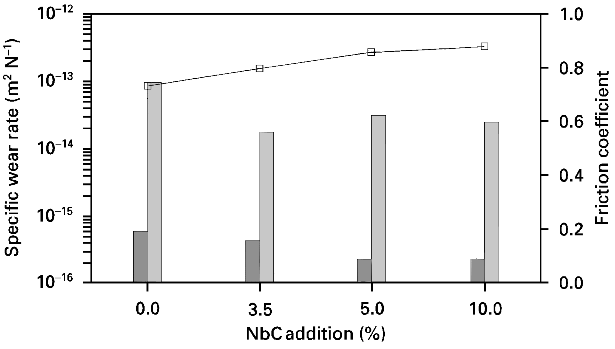

Figure 9 Wear specific rates and friction coefficient of sintered materials. (▨), pin; (▤), disc; (—⊟—), Friction coefficient.

Figure 10 Wear tract of tested metal matrix composites.

materials quenched from 950 °C, with Rockwell C hardness values between 52 and 58 HRC, and some maximum values between 62 and 67 HRC. These are lower than those of wrought HSSs but are in agreement with the density levels and are in the same range as those obtained by Dubois *et al.* [18].

In Fig. 2, the transverse rupture strength of the materials as a function of the austenitizing temperature can be analysed. As with hardness, this property is highly affected by the low level of density of the materials. Conventional wrought HSS exhibits bending strengths four or five times higher [19], but the materials under study exhibit similar mechanical behaviour to those produced by the same manufacturing route by Kothari [20].

The materials quenched from 950 °C were then tempered four times at 550 °C for 120 min. The third and fourth tempers were made to study the influence of

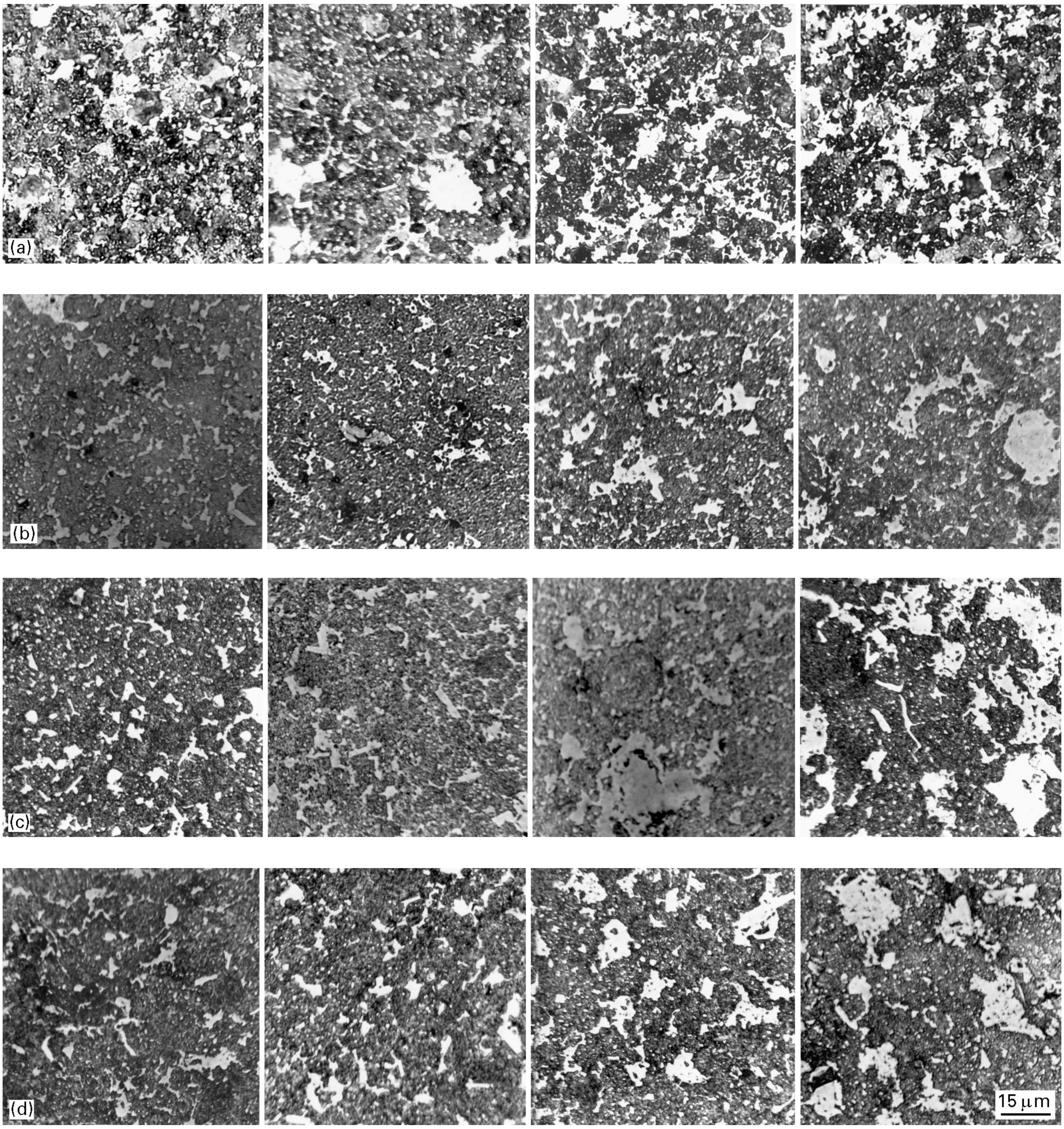

Figure 11 Microstructures of materials in different states: (a) after sintering; (b) after quenching; (c) after second tempering; (d) after fourth tempering.

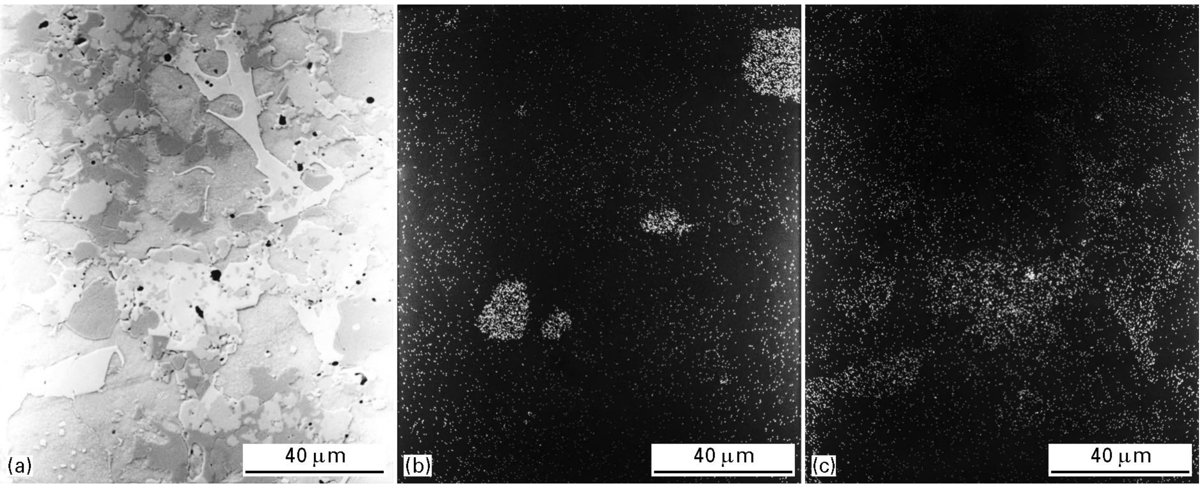

Figure 12 (a) Microstructure of 5% NbC reinforced HSS; (b) mapping of Cu; (c) mapping of W.

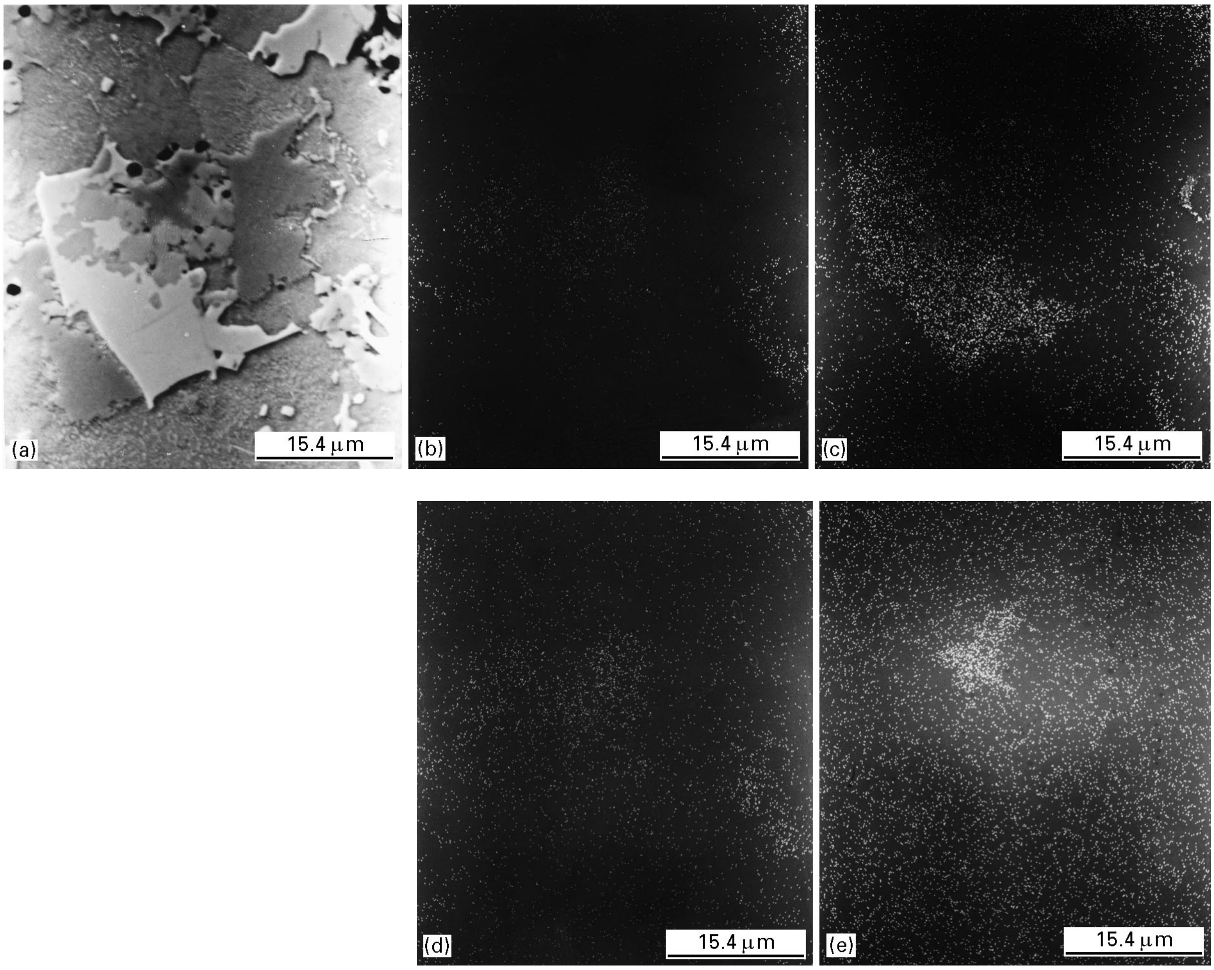

Figure 13 (a) Microstructure of 5% NbC reinforced HSS; (b) mapping of Nb; (c) mapping of W; (d) mapping of V; (e) mapping of Cr.

niobium carbide and the possibility of its re-precipitation at tempering temperature and the effect on secondary hardness, even though the dilatometric study shows that the main structural changes occur only in the first tempering. Figs 3–5 are of the dilatometric study. It is confirmed for this material that sintering temperatures below 1100 °C can be achieved. The dilatometric study shows the same points of temperature, at which liquid-phase sintering is produced, as those reported by Bolton and Baah [21] obtained through differential thermal analysis. It also shows how a second tempering does not produce any structural transformation and in consequence could be avoided (Figs 4 and 5) if its positive influence on bending strength and toughness is not a consideration.

Figs 6 and 7 display the hardnesses and bending strengths of heat-treated materials in each treatment condition. Tempering does not much improve hardness, but its influence on bending strength is positive, in comparison with the results of quenched materials.

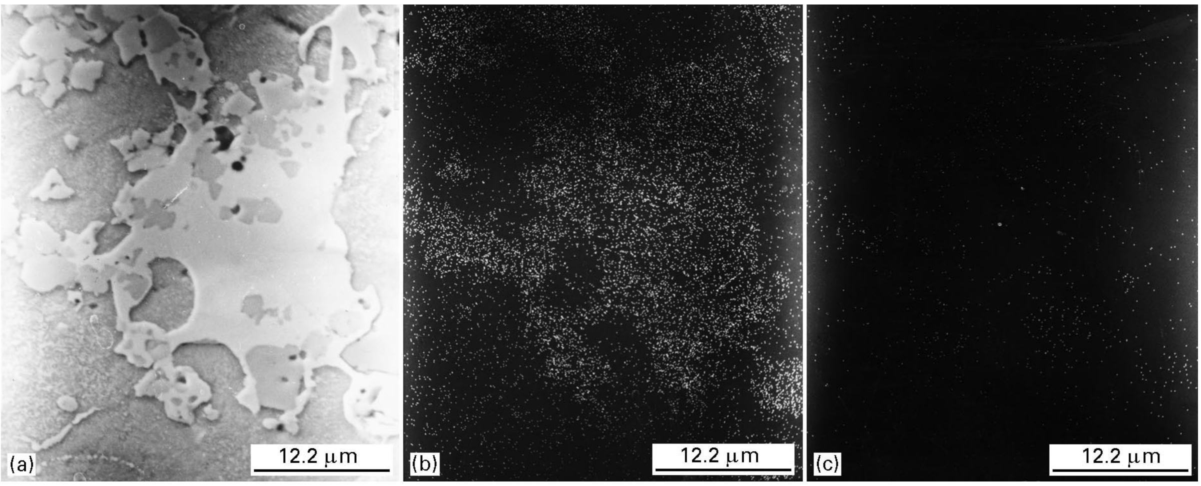

Figure 14 (a) Microstructure of 5% NbC reinforced HSS; (b) mapping of Mo; (c) mapping of Nb.

The best materials from the viewpoint of mechanical properties were tested for wear properties as shown in Fig. 9 where the specific wear rates of the pin and disc are compared with the friction coefficient. The friction coefficient increases with increasing amount of reinforcement. It might be expected that the reinforcement content should decrease the specific wear rate, but the high density of the NbC produces an increase in that value due to the large amount of material lost when a carbide is removed. Fig. 9 suggests that, from the viewpoint of wear behaviour, the 3.5% reinforcement is the optimal composition. If the wear track is examined (Fig. 10), the bond between matrix and reinforcement is seen to be strong because carbide removal is hardly apparent. The best material is that with the 3.5% NbC addition but the least hard material (the unreinforced material) produces the highest wear in the counterpart. This could be due to the wear mechanism of this material, which is highly dependent on the carbide size and its cohesion with the steel matrix, and the unreinforced M2 has the smallest carbide size of all the materials under study, all of which are primary carbides with the highest cohesion level possible (and, as the reinforcement is increased, the carbide coalescence is higher and in consequence the size of the carbides increases).

All the materials exhibit very similar microstructures (Fig. 11): thin carbides in a martensitic–bainitic matrix. In some areas, agglomerations of carbides can be seen as well as some phosphorus eutectic such as those verified in [21]. The carbide agglomerations observed are due to the non-assisted system used for mixing the powders; they could be avoided by using a more efficient mixing system such as a ball mill. The microstructural nature of the constituents was established through a microhardness study (Fig. 8) which shows that, in the sintered and tempered state, bainite appears and, in the quenched state, martensite. The scanning electron microscopy study (Figs 12–14) shows that V and Cr are well distributed in the M2 matrix (Fig. 13). W and Mo are mainly concentrated in carbides of M_2C and M_6C types, as well as the Nb, which is seen agglomerated by W and Mo carbides (Figs 13 and 14). This provides the good bond between the reinforcement and the matrix and, in consequence, good wear behaviour. In addition, Cu is segregated in some parts (Fig. 12) but is otherwise well distributed in the M2.

5. Conclusions

1. The compacting pressure of 700 MPa used in this work gives densities of M2 with copper–phosphorous additives close to the 93% of the theoretical density.
2. Using additions of copper–phosphorous, M2 reinforced with NbC can be sintered at 1150 °C or at lower temperatures in a N_2–H_2 atmosphere.
3. The best results were obtained at 950 °C for austenitizing.
4. Heat treatments produce a decrease in bending strength in the sintered and quenched conditions. In all cases, a higher percentage of additives impairs ductile behaviour.
5. A dilatometric study allows the first tempering to produce the main microstructural changes; so further tempers produce no substantial changes in mechanical properties.
6. The reinforcement of M2 with NbC improves the wear behaviour of this HSS. The best proportion of reinforcement is 3.5%.

Acknowledgements

This work was carried out with the financial support of the Instituto Español de Cooperación Iberoamericana. The authors wish to thank Dr L.E.G. Cambronero and Mr F. Velasco for their help in the research.

References

1. C. S. WRIGHT, J. D. BOLTON, N. M. ECBBECK and A. WLONSKI, in Proceedings of the First International Conference on High Speed Steels, Loeben, 26–28 March (1990).
2. J. M. TORRALBA, L. E. G. CAMBRONERO, J. M. RUIZ-PRIETO and N. M. DOS NEVES, *Powder Metall.* **36** (1993) 55.

3. R. A. QUEENEY, *Adv. Powder Metall. Particulate Mater.* **2** (1992) 355.
4. I. M. MARTINS, M. M. OLIVEIRA and H. CARVALHINHOS, in Colloquium on Controlling the Properties of Powder Metallurgy Parts through their Microstructure, Paris, 19–21 March 1990 (1990).
5. B. P. SAHA and G. S. UPADHYAYA, *Powder Metall. Int.* **24** (1992) 6.
6. D. J. BELTZ, Metal Powder Rep. **3** (1988) 193.
7. C. JOUANNY-TRESY, PhD thesis, Ecole Nationale Superieure des Mines de Paris, Paris (1992).
8. M. IGHARO and J. V. WOOD, *Powder Metall.* **33** (1990) 313.
9. L. E. G. CAMBRONERO, J. M. TORRALBA and J. M. RUIZ-PRIETO, *Adv. Powder Metall. Particulate Mater.* **3** (1992) 205.
10. E. DUDROVA, in Proceedings of the Eighth International Conference on Powder Metallurgy in CSFR (Czechoslovaquian Republic) 1992 (1992) 209.
11. J. D. BOLTON, M. JEANDIN and C. JOUANNY-TRESY, in proceedings of the International Conference on Powder Metallurgy, London **2** (1990) 265.
12. W. C. ZAPATA and C. E. DA COSTA, in III Seminario de Metalurgia do Pó, ABM, Sao Paulo, 23–25 October 1991 (1991).
13. T. BELL, *Powder Metall.* **34** (1991) 253.
14. G. GREETHAM, British Patent GB 2187663A (1986).
15. M. COLOMB and G. P. BRUNEL, French Patent 8604309 (1986).
16. C. JOUANNY-TRESY, M. VARDAVOULAS and M. JEANDIN, *J. Mater. Sci.* **28** (1993) 6147.
17. W. C. ZAPATA and C. E. DA COSTA, in IBEROMET II, Mẽxico, 8–14 November 1992 (1992).
18. P. H. DUBOIS, L. THEMELIN and G. P. BRUNEL, in Colloquium on Controlling the Properties of Powder Metallurgy Parts through their Microstructure, Paris, 19–21 March 1990 (1990).
19. P. BEISS, R. WÄHLING and D. DUDA, *Mod. Dev. Powder Metall.* **17** (1985) 331.
20. N. C. KOTHARI, in Proceedings of the Fifth International Symposium on the Science and Technology of Sintering, Vancouver, 23–26 July (1991) 681.
21. J. D. BOLTON and H. O. BAAH, *Powder Metall.* **34** (1991) 273.

Received 22 February 1996
and accepted 18 March 1998