# Effect of Carbide Addition on Sintering of M2 Tool Steel

Haorong Zhang, Donald F. Heaney, and Randall M. German

Center for Innovative Sintered Products
P/M Lab, 118 Research West
The Pennsylvania State University
University Park, PA 16802-6809

## ABSTRACT

In this present investigation tungsten carbide powder was mixed with PIM grade M2 tool steel powder and formed by pressing. The sintering densification, sintered properties, and microstructure are examined for the specimens of M2 tool steel with and without WC additions. The additions of the carbide effectively widened the processing window due to the additional supersolidus liquid produced by the reaction of WC with the matrix metals. Compacts with WC additions can be sintered to full density without distortion in either nitrogen or vacuum, at temperature from 1240°C to 1250°C. Specimens without WC addition can't be sintered to high density at temperature of 1240°C and warped when sintering in nitrogen. They can only be sintered to high density in vacuum at 1250°C. Sintered density and hardness of the specimens with the carbide additions were significantly improved and more uniform when compared to the same M2 tool steel powder without carbide addition.

## INTRODUCTION

M2 tool steel has been used to replace T1 tool steels in some application since 1950's. This is a result of lower cost, greater toughness at the equivalent hardness, better wear resistance, and higher hot hardness. Traditionally, the steel has been produced via casting and hot working.(1,2) In cast components segregation and non-uniform carbide distribution often degrade its mechanical properties and cutting performance. Powder metallurgy and powder injection molding (PIM) technologies have overcome these problems, and produce finer and more uniform microstructures, which improve the mechanical properties, grindability, and cutting performance.(3) However, the processing temperature window for M2 tool steel powder is small and a tightly controlled process is required to produce quality product.

Without close processing control, sintering densification of M2 tool steel with precise dimension control proves difficult, because the densification is based on supersolidus liquid phase sintering.(4,5) Traditional liquid-phase sintering relies on a mixture of two or more powders to form a liquid phase between the particles at the sintering temperature. The amount of liquid phase can be controlled by adjusting the powder mixing ratio. While supersolidus liquid-phase sintering takes advantage of prealloyed powders to create a situation where liquid forms inside the particles. When the prealloyed powders are heated to the partially molten condition they exhibit rapid densification.(6,7,8) The liquid forms within the particles, causing each particle to fragment into individual grains. Subsequent repacking of the fragments caused by the capillary force from the wetting liquid results in rapid densification. Unlike traditional liquid-phase sintering, the amount of liquid and the temperature, at which the liquid phase appears, depend on the exact composition and processing cycle. To achieve good sintering result, precise process control is essential. The sintering temperature should fall between the solidus and liquidus. Temperatures above solidus line give mushy particles that flow once sufficient liquid forms along the grain boundaries. The viscosity of the solid-liquid mixture decreases as the liquid volume fraction increases, so more liquid makes for faster sintering but for less dimensional precision. Consequently, temperature is a main determinant of sintered density and precision.

The solidus-liquidus separation of M2 tool steel at carbon contents between 1.0% to 1.2% is nearly constant, about 13°C. Sintering above the liquidus temperature results in slumping, while sintering below solidus temperature results in poor densification. Too long a sintering time or too much liquid phase will also cause grain growth and carbide coarsening.

Carbon control is also critical in the processing of M2 tool steel.(5) This is because the achievable volume of the liquid phase and the liquidus and solidus temperature during sintering are directly correlative to the carbon content in the steel. According to German's computer model,(9) the relation between the carbon content and the liquidus and solidus temperatures can be determined by the following equations:

$$T_s = T_{so} + BX_c \qquad (1)$$

$$T_l = T_{lo} + AX_c \qquad (2)$$

Where $X_c$ = weight fraction of carbon
$T_s$ = solidus temperature
$T_l$ = liquidus temperature
$T_{so}$ = solidus temperature extrapolated to 0 carbon content
$T_{lo}$ = liquidus temperature extrapolated to 0 carbon content
$A$ = liquidus slope
B = solidus slope

Accordingly, at 1.0 % carbon, the onset densification temperature would be 1239°C and at 1.2% carbon it would be 1219°C. Since the sintering window is only 13°C, this 20°C sintering temperature change with a slight carbon shift makes processing difficult.

Many factors can contribute to the carbon fluctuations in M2 tool steel. The surface oxide in the powder may remove carbon during sintering. If the oxygen content in the powder is in excess of a certain level, or if the oxide is not properly reduced before sintering, the components are more likely to crack or distort during sintering.(5) The blending of graphite into the water atomized M2 steel powder to balance the excessive oxide has been reported.(10) This results in improved properties. On the other hand, the binder in injection molded parts may leave behind residual carbon during debinding and cause carbon over saturation. Too much carbon may result in melting and slumping during sintering. Imprecise carbon control and uneven carbon distribution are the common problems in processing of tool steel powders.

Previous studies (11-21) have demonstrated that M-class tool steel can be made more wear resistance than cast-forged material with the same composition by dispersion of hard particles into the matrix. In some examples, abrasive wear resistance was increased by an order of magnitude by the addition of 10 wt% alumina.(21)

The objective of the present investigation was to widen the sintering window, obtain precise dimension control in sintering of M2 tool steel, and to enhance the properties of the press and sintered and powder injection molded M2 tool steel by the addition of tungsten carbide powder. The tungsten carbide is expected to improve the uniformity of the liquid phase distribution during sintering, to supplement the carbon source, and to strengthen the steel.

## EXPERIMENT

The gas atomized M2 tool steel powder was produced by Ultrafine Powder Technology. The pycnometer density of the powder was measured as 8.02 g/cm$^3$. The particle size and the nominal chemical composition of the powder are listed in Tables 1 and 2.

Table 1: Nominal Chemical Composition of the M2 Tool Steel Powder.

| Element | W | Mo | Cr | V | C | Si | O | S | N | Fe |
|---|---|---|---|---|---|---|---|---|---|---|
| Wt.% | 6.08 | 5.16 | 4.10 | 1.93 | 0.80 | 0.18 | 0.037 | 0.018 | 0.005 | Bal. |

Table 2: Particle Size Distribution of the M2 Powder.

| Micrometer | 5.7 | 11.2 | 18.6 |
|---|---|---|---|
| Cum.% | 10 | 50 | 90 |

The tungsten carbide powder was WC 50 from Smith Tool. The pycnometer density of the carbide powder was measured as 15.65 g/cm$^3$. The particle size distribution is listed in Table 3.

Table 3: Particle Size Distribution of the Tungsten Carbide Powder.

| Micrometer | 13 | 26 | 44 |
|---|---|---|---|
| Cum.% | 10 | 50 | 90 |

The mixing ratios of WC powder to M2 tool steel powder were 1% increments from 0 to 6%. Each of the powder combinations was mixed in a Turbula for 30 minutes. To avoid segregation and to facilitate the pressing, the mixed powders had 3wt% polymer-wax binder added and were granulated to –80 mesh size prior to die pressing into tensile bars under a pressure of 320 MPa.

Before sintering, the tensile specimens were presintered at 800°C for 1 h in hydrogen gas with the heating rate of 10°C/min. This removed the binder and reduced surface oxides. The presintered specimens were either sintered in a CM high temperature tube furnace or a graphite vacuum furnace with vacuum level of 0.3 Pa. According to German's model the onset densification temperature of the M2 tool steel with 0.8% carbon is around 1245°C. The sintering window is 13°C. Therefore the sintering temperatures were selected at 1240°C, which is below the densification temperature, and 1250°C, which is inside the sintering window. The sintering atmospheres were vacuum and nitrogen plus 5% hydrogen. The sintering schedules are listed in Table 4.

Table 4: Sintering Schedules.

|  | Heating Rate, °C/min, 0-1200°C | Heating Rate, °C/min, >1200°C | Holding Temp. | Holding Time | Atmosphere |
|---|---|---|---|---|---|
| 1 | 10 | 5 | 1250°C | 60 min | $N_2$+5%$H_2$ |
| 2 | 10 | 5 | 1250°C | 30 min | Vacuum |
| 3 | 10 | 5 | 1240°C | 30 min | Vacuum |

The sintered specimens were examined for density, hardness, tensile strength, and microstructure. The theoretical densities of M2 tool steel with WC additions were calculated based on theoretical density of M2 tool steel at 8.10 g/cm$^3$ and of WC at 15.7 g/cm$^3$. The microscopy specimens were etched with 6% nital. The density and tensile strength were averaged from three specimens for each group. The hardness was averaged from three specimens with three repeats on each specimen for each group. The carbon analysis was only carried out on the powder and on one each of the vacuum sintered and nitrogen + 5% hydrogen sintered specimen of M2 tool steel without WC content. The cross head speed in tensile testing was 6.35 mm/min.

## RESULTS

All specimens with WC additions sintered equally well in both nitrogen and 5% hydrogen or in vacuum with respect to shape retention. The specimens without WC were warped when sintered in nitrogen and 5% hydrogen, while those sintered in vacuum retained shape, as shown in Figure 1.

The density of the specimens sintered in vacuum at 1240°C increased with the increase in WC content, and reached full density when the WC content reached 6 wt%, as shown in Table 5. Full density was achieved in all specimens with or without WC content at a sintering temperature of 1250°C in vacuum. However, the hardness increased, but tensile strength decrease with increasing WC content, as shown in Table 6.

The carbon content in the original M2 tool steel powder was 0.80%. The specimen of M2 steel without WC content and sintered in vacuum had a carbon content of 0.79%. The specimen of

M2 steel without WC sintered in nitrogen and 5% hydrogen had a carbon content of 0.78%. The specimen without WC addition sintered in nitrogen and 5% hydrogen at 1250°C reached 95.6% density and warped. While those with more than 3% WC addition sintered to near full density without warping under the same conditions, as shown in Table 7.

Table 5: Properties of M2 Tool Steel Sintered in Vacuum at 1240°C for 30 min.

| Specimen | Density | % theoretical | Tensile strength, MPa | HRC |
|---|---|---|---|---|
| M2 | 6.43 | 79.4% | 655 | 12.3 |
| M2+1%WC | 7.00 | 86.0% | 865 | 26.0 |
| M2+2%WC | 7.35 | 89.5% | 991 | 33.1 |
| M2+3%WC | 7.55 | 92.0% | 909 | 38.7 |
| M2+4%WC | 8.13 | 98.5% | 1056 | 48.1 |
| M2+6%WC | 8.32 | 99.9% | 1135 | 52.4 |

Table 6: Properties of M2 Tool Steel Sintered in Vacuum at 1250°C for 30 Min.

| Specimen | Density | % theoretical | Tensile strength, MPa | HRC |
|---|---|---|---|---|
| M2 | 8.09 | 99.9% | 1305 | 43 |
| M2+1%WC | 8.10 | 99.5% | 1346 | 48 |
| M2+4%WC | 8.24 | 99.8% | 1260 | 50 |
| M2+6%WC | 8.32 | 99.9% | 1134 | 55 |

Table 7: Properties of M2 Tool Steel Sintered at 1250°C in $N_2$ + 5%$H_2$ for 60 min.

| Specimen | Density | % theoretical | Tensile strength, MPa | HRC |
|---|---|---|---|---|
| M2 | 7.75 | 95.7% | N/A | 40 |
| M2-1% WC | 7.76 | 95.3% | 756 | 43 |
| M2-2%WC | 8.00 | 97.8% | 966 | 52 |
| M2-3%WC | 8.04 | 98.1% | 976 | 58 |
| M2-4%WC | 8.24 | 99.8% | 1025 | 58 |
| M2-5%WC | 8.29 | 99.9% | 1128 | 57 |
| M2-6%WC | 8.32 | 99.9% | 1124 | 57 |

Optical microscopy revealed that the liquid phase did not appear in the M2 tool steel specimens during sintering at 1240°C in vacuum, and the specimen remained very porous, as shown in Figure 2. The M2 + 6% WC specimen was sintered to full density at 1240°C in vacuum, as shown in Figure 3. Figures 4 and 5 show the microstructure of M2 + 6% WC and M2 sintered in vacuum at 1250°C for 30 minutes. Both materials were sintered to near full density, however, there is more liquid phase in the M2 + 6% WC specimen. The microstructure of M2 steel sintered in nitrogen and 5% hydrogen at 1250°C is not uniform, as shown in Figure 6, which might be the cause of distortion. In comparison, the specimens of M2 + 6% WC sintered in nitrogen and 5% hydrogen at 1250°C were near fully densified, and the microstructure is homogeneous, as shown in Figure 7.

## DISCUSSION

The addition of tungsten carbide to M2 steel widened the sintering window. The tungsten carbide in the M2 tool steel serves as an additional carbon source, which facilitates carbon control and sintering shrinkage. The tungsten carbide also improves hardness, and possibly the wear resistance.

The M2 powder was used in this study had a carbon content of 0.80%. The onset densification temperature for M2 tool steel with this carbon level should be around 1245°C according to German's model. That is why the steel without WC addition sintered to only 79.4% of theoretical density at 1240°C in vacuum. However, at the same sintering temperature in vacuum, the sintered density increased with increasing amounts of tungsten carbide as shown in Table 5. Another effect of tungsten carbide on the sintering density is due to the formation of a liquid phase in the areas adjacent to the WC particles, as shown in Figure 3.

At 1250°C in vacuum, all the specimens with and without tungsten carbide addition sintered to near full density. The hardness increased, however, the tensile strength decreased with the increasing tungsten carbide content, as shown in Table 6. That result indicates a role of tungsten carbide in hardening the matrix, but the brittle carbide phase formed by reaction of tungsten carbide with steel matrix reduced the tensile strength.

When sintered in nitrogen and 5% hydrogen, the M2 tool steel without WC addition reached to 95.7% of theoretical density and became seriously warped. This is likely due to the uneven liquid phase distribution because of carbon loss during sintering. But all the specimens with WC addition sintered in nitrogen and 5% hydrogen maintained shape. The density increased with increasing amount of WC addition, and reached 99.8% when the WC content was higher than 4%, as shown in Table 7. In this case the liquid phase around the tungsten carbide particles helped densification and prevented distortion.

## SUMMARY

The sintering of components from M2 tool steel powder with precise dimension control is sometimes difficult. That is mostly due to the nature of the supersolidus liquid phase sintering process. The sintering window is usually very narrow. The amount of the liquid phase produced during sintering is not constant, because it depends not only on temperatures but also on the carbon level. So any discrepancy in temperature and carbon level will result in distortion, inadequate densification, or oversintering. The addition of tungsten carbide widens the sintering window and makes the achievable liquid volume more constant. Consequently, it makes the process easier to control. The tungsten carbide also improves the hardness and most likely the wear resistance of the sintered M2 steel. However, the strength decreases slightly. In this study 4 to 6% of tungsten carbide in M2 steel yielded the best results.

## ACKNOWLEDGEMENTS

The research was supported by Center for Innovative Sintered Products. The authors wish to thank Kristina Cowan and Louis Campbell for their helps in the microstructure examination and particle size analysis.

## REFERENCES

1. G. Hoyle, *High Speed Steel*, Butterworths, Boston, MA, 1988.
2. N. Myers and R. M. German, "Supersolidus Liquid Phase Sintering of Injection Molded M2 Tool Steel," *Advances in Powder Metallurgy and Particulate Materials*, vol. 3, 1997, Metal Powder Industries Federation, Princeton, NJ, pp. 18.77-18.90.
3. H. E. Amaya and E. R. Andreotti, "Production of M2 Tool Steel Blanks by MIM," *Advances in Powder Metallurgy*, 1990, vol. 3, Metal Powder Industries Federation, Princeton, NJ, pp. 385-399.
4. T. Kirk, H. Zhang, S. Caldwell and J. Oakes, "Carbon Control in PIM Tool Steel," *Advances in Powder Metallurgy and Particulate Materials*, 1995, vol. 2, Metal Powder Industries Federation, Princeton, NJ, pp. 6.199 - 6.204.
5. H. Zhang, "Carbon Control in PIM Tool Steel," *Mater. Manuf. Process*, 1997, vol.12, pp. 673 - 679.
6. R. M. German, *Sintering Theory and Practice*, John Wiley and Sons, New York, NY 1996, p. 374.
7. R. M. German, "Densification of Prealloyed Tool Steel Powders: Sintering Model," *Inter. J. Powder Meta.*, 1997, vol. 33, No.6, pp. 49 – 61.
8. R. M. German, "Advances in High Alloy Sintering Using Supersolidus Liquids," *Advances in Powder Metallurgy and Particulate Materials*, 1997, vol. 2, Metal Powder Industries Federation, Priceton, NJ, pp. 14.3 – 14.18.
9. R. M. German, "Computer Model for the Sintering Densification of Injection Molded M2 Tool Steel," *Inter. J. Powder Meta.*, 1999, vol. 35, No. 4, pp. 57 - 67.
10. D. W. Hetzner, J. A. Laverick, and D. G. Hennessy. "Effect of Sintering Temperature on Carbide Distribution in M2 P/M Tool Steel," *Advances in Powder Metallurgy*, 1989, vol. 2, Metal Powder Industries Federation, Princeton, NJ, pp. 311 - 326.
11. Y. Kim, H. Chung, B. K. Kim, and J. H. Ahn, "Process and Properties of Mechanically Alloyed High Speed Tool Steels," *Advances in Powder Metallurgy*, 1992, vol. 7, Metal Powder Industries Federation, pp. 383 - 396.
12. J. R. Howell, A. Lawley, and R. D. Rivers, "Low Cost Wear-Resistant Composites Via Injection Molding of Pliable Powders," *Horizons of Powder Metallurgy*, Part II, W. A. Kaysser and W. J. Huppman (eds.), Verlag Schmid, Freiburg, Germany, 1986, pp. 641- 644.
13. G. S. Upadhya and P. K. Kar, "Liquid Phase Sintering of TiC or TiN Enriched T15 Grade High Speed Steels and Their Mechanical Properties," *Sintering '91*, A. C. D. Chaklader and J. A. Lund (eds.), Trans Tech Publ., Brookfield, VT, 1992, pp. 479 - 494.
14. B. P. Saha and G. S. Upadhyaya, "Liquid Phase Sintering of T15 and T42 High Speed Steel Composites Containing Ti(C,N)," *Powder Met. Inter.*, 1992, vol. 24, pp.345-350.
15. J. D. Bolton, M. M. Oliveira, "Sintering Behaviour of High Speed Steel Metal Matrix Composites Containing TiC and TiN Ceramic Additions," *Proceedings 1993 Powder Metallurgy World Congress, Part 1*, Y. Bando and K. Kosuge (eds.), Japan Society Powder and Powder Metallurgy, Kyoto, Japan, 1993, pp. 369 - 372.

16. M. M. Oliveira and J. D. Bolton, "Sintering of M3/2 High Speed Steel Modified by Additions of Copper Phosphide and Titanium Based Ceramic Compounds," *Powder Met.*, 1995, vol. 38, pp. 131 - 140.
17. H. Miura, T Baba, N. Nagatani, Y. Kawakami, and A. Ishibahi, "Development of High Performance Ferrous Abrasive Wear Resistant Materials Through MIM Process," *J. Japan Soc. Powder Powder Met.,* 1996, vol. 43, pp. 1333 - 1338.
18. M. Oliverira and J. D. Bolton, "Effect of Ceramic Particles on the Mechanical Properties of M3/2 High Speed Steel," *Inter. J. Powder met.,*1996, vol. 32, pp. 37- 49.
19. J. D. Bolton and A. J. Grant, Microstructural Development and Sintering Kinetics in Ceramic Reinforced High Speed Steel Metal Matrix Composites," *Powder Met.*, 1997, vol. 40, pp. 143 - 151.
20. H. Miura, H. Morikawa, Y. Kawakami, and A. Ishibashi, "Development of High Performance Sliding Abrasive Wear Resistant Materials Through Powder Injection Molding," *Advances in Powder Metallurgy and Particulate Materials,* 1998, Metal Powder Industries Federation, Princeton, NJ, 1998, pp. 5.183 - 5.191.
21. R. A. Queeney, J. L. Miller, R. J. Beltz, and J. D. Dankoff, " Fracture Resistance of $Al_2O_3$ Reinforced High Speed Steel," *Advances in Powder Metallurgy,* 1989, vol. 2, Metal Powder Industries Federation, NJ, pp. 345 -354.

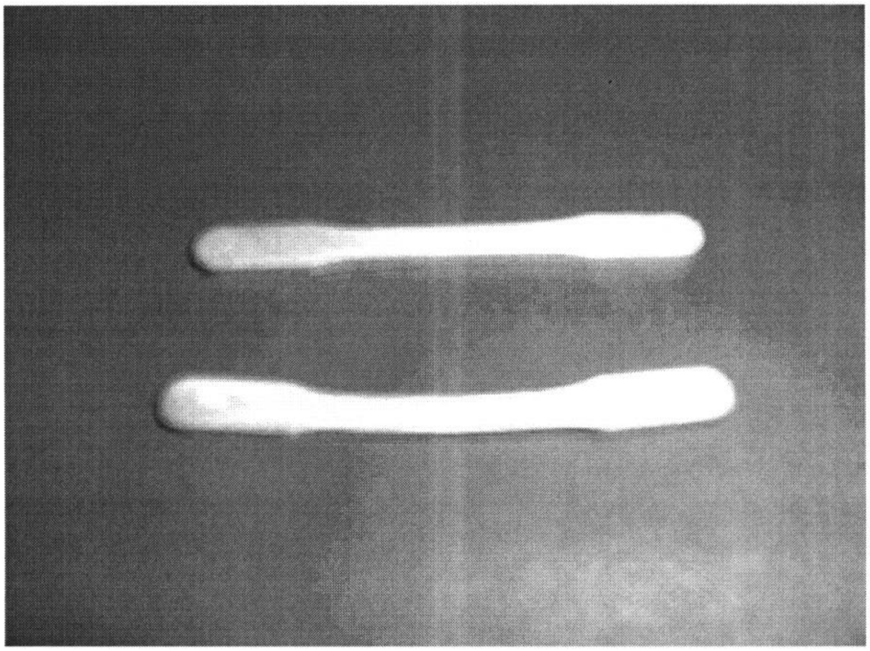

Figure 1: Specimens sintered in $N_2 + 5\% H_2$ at 1250°C for 1 hour:
Top: M2 tool steel+6% WC; Bottom: M2 tool steel

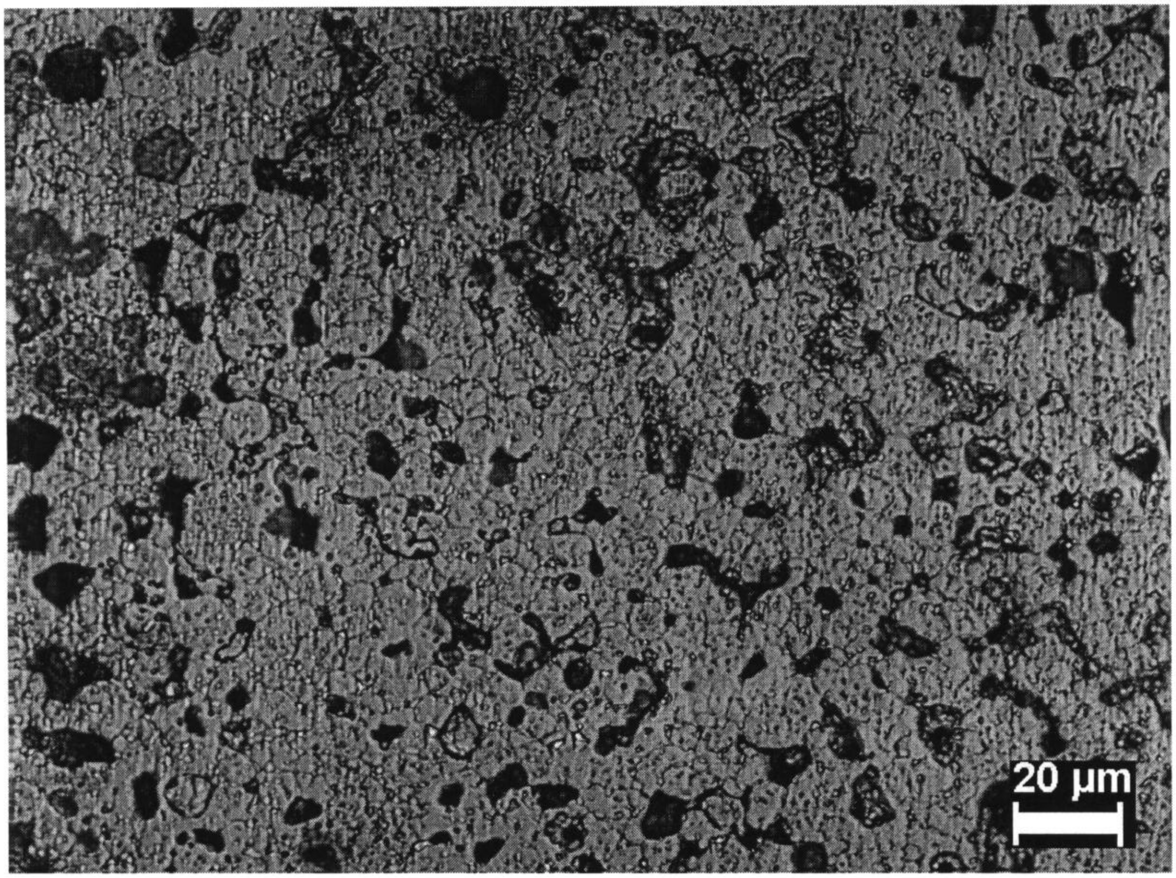

Figure 2: M2 tool steel sintered in vacuum at 1240°C for 30 minutes.

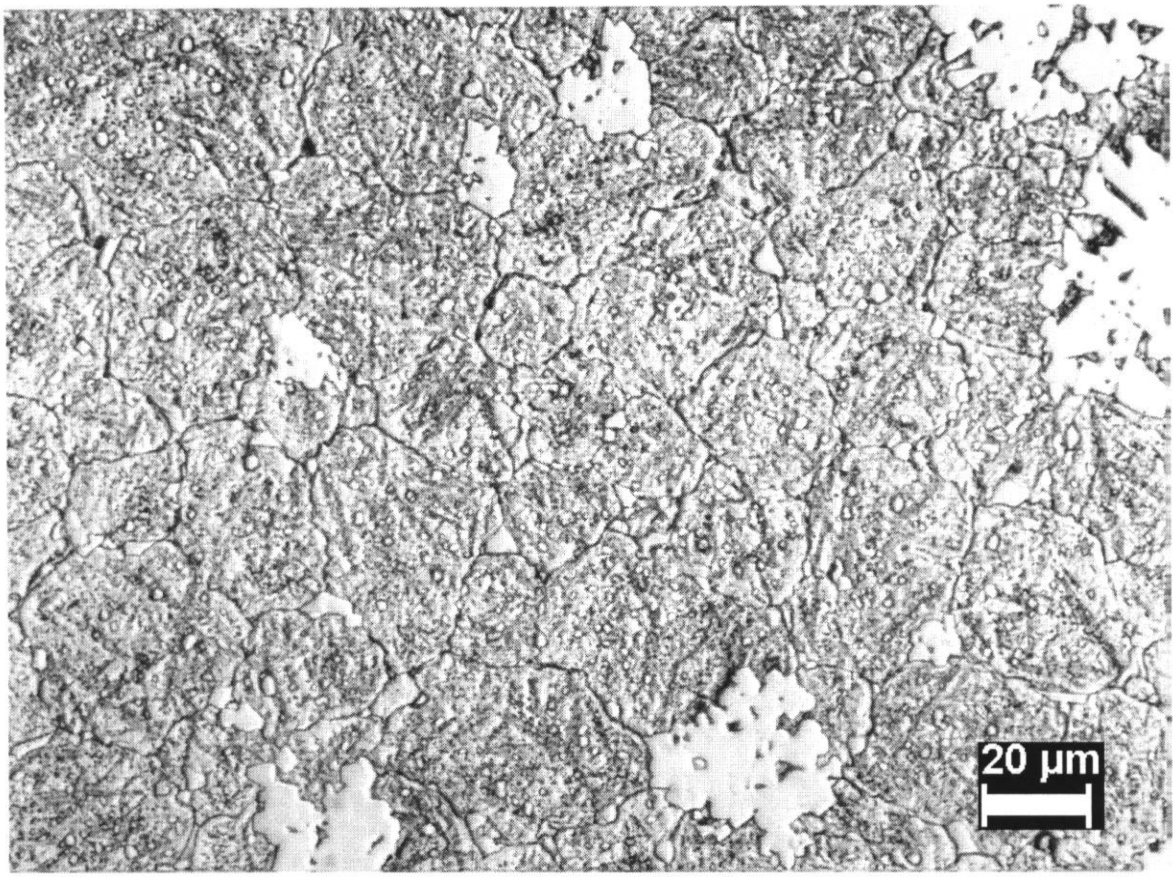

Figure 3: M2 tool steel + 6% WC sintered in vacuum at 1240°C for 30 minutes.

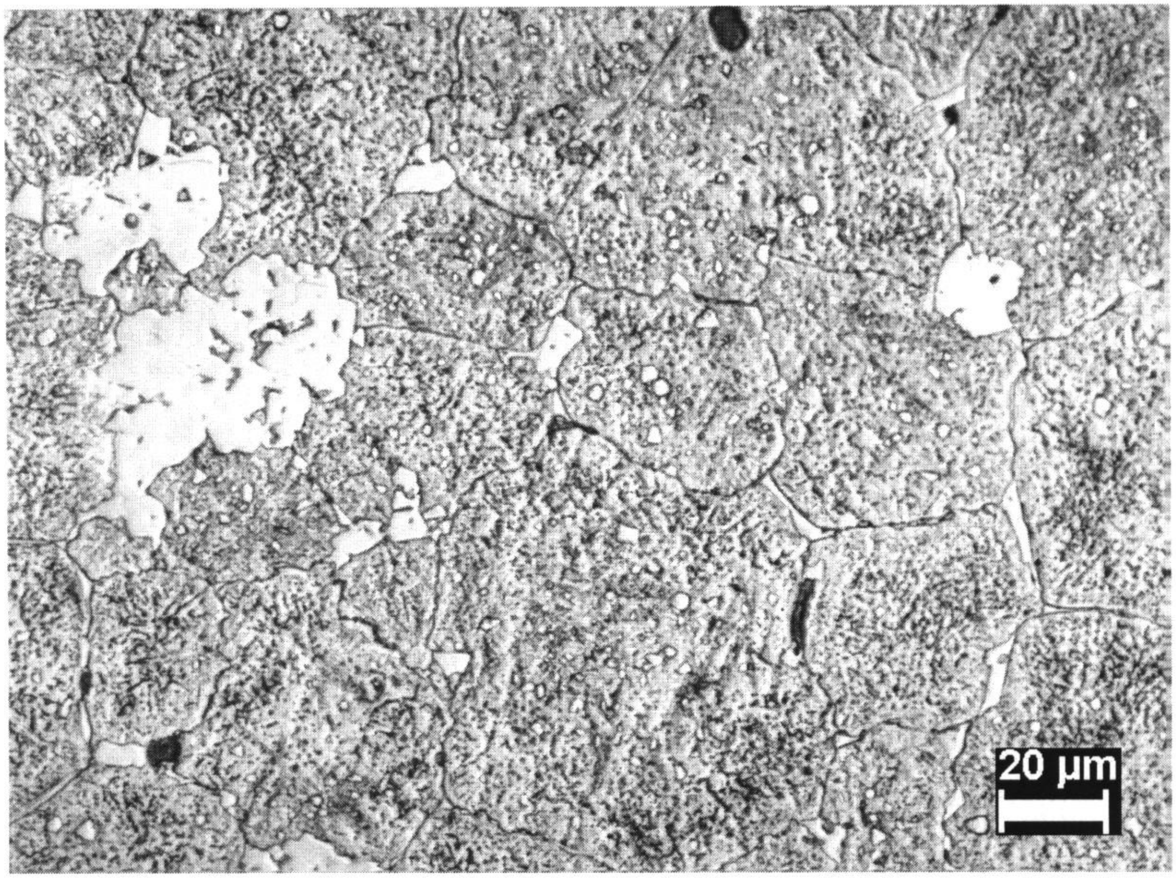

Figure 4: M2 tool steel + 6% WC sintered in vacuum at 1250°C for 30 minutes.

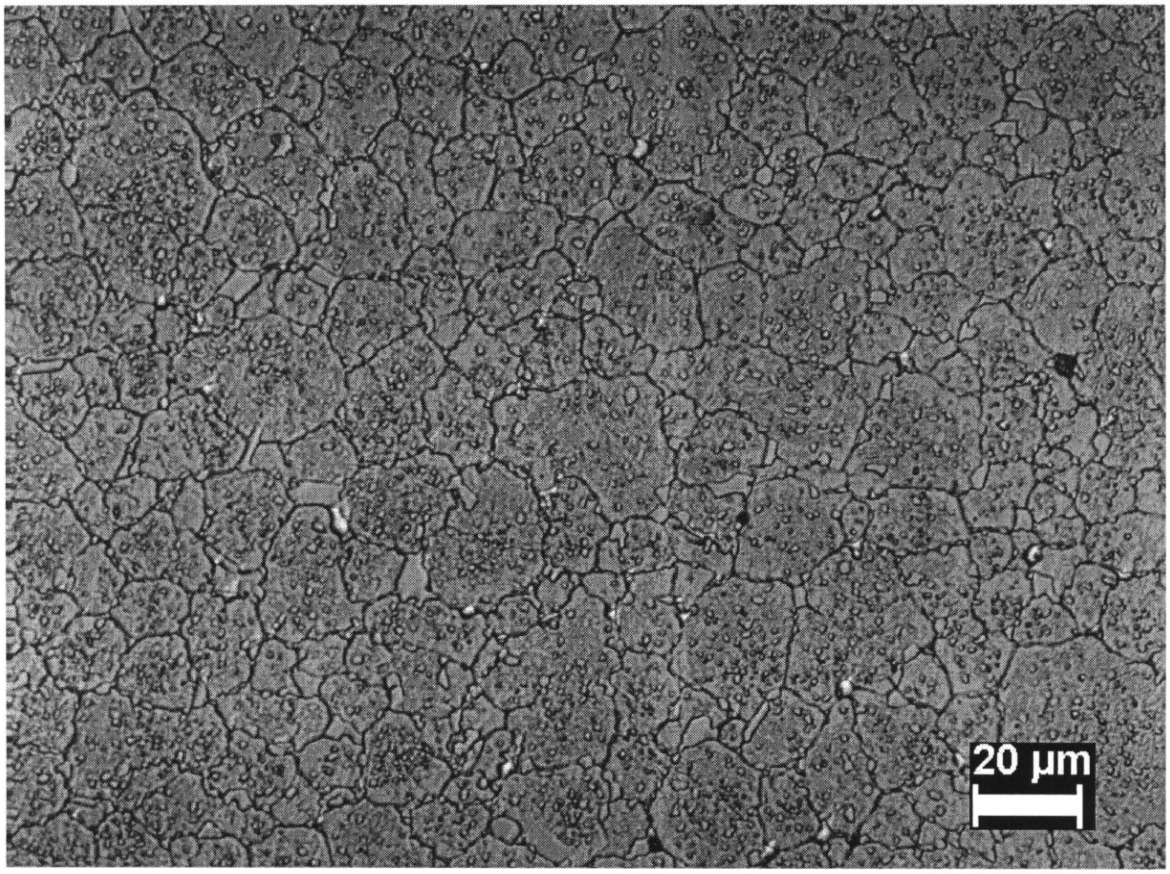

Figure 5: M2 tool steel sintered in vacuum at 1250°C for 30 minutes.

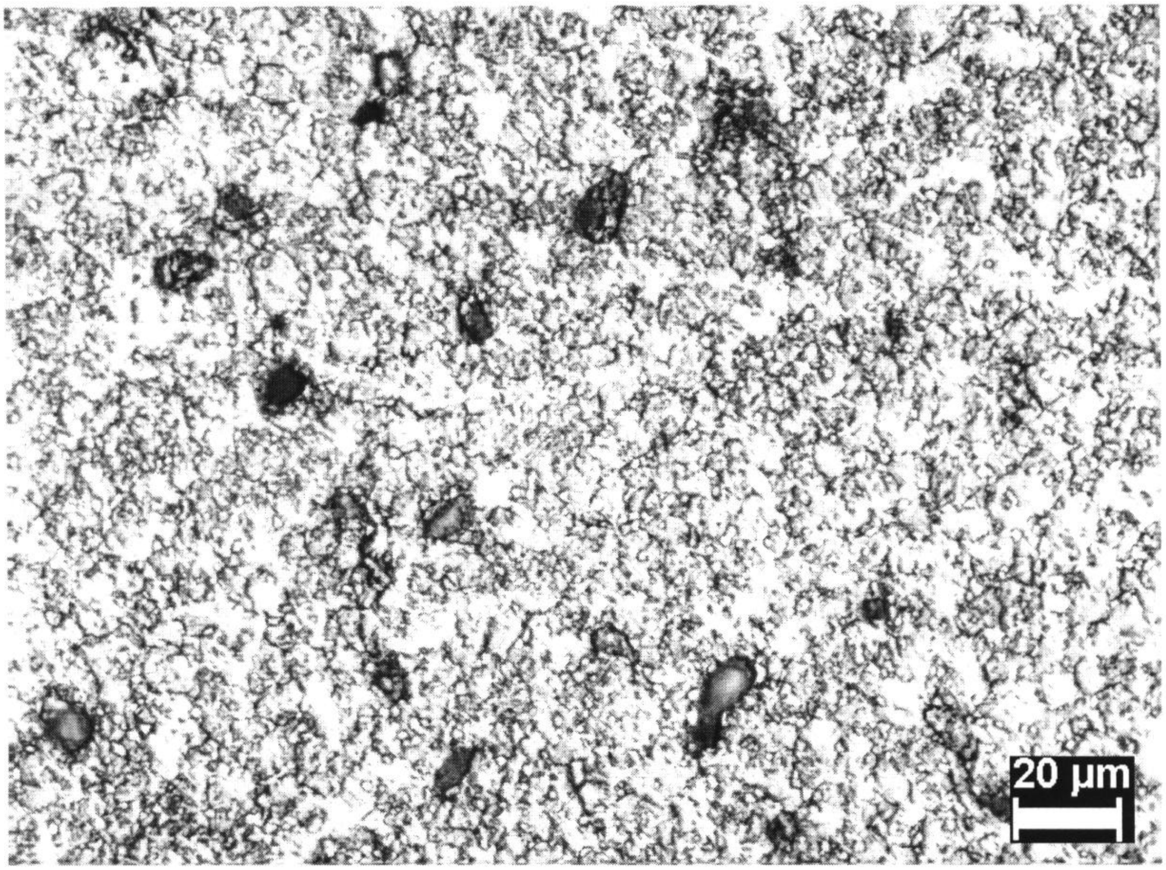

Figure 6: M2 tool steel sintered in $N_2$ + 5% $H_2$ at 1250°C for 60 minutes.

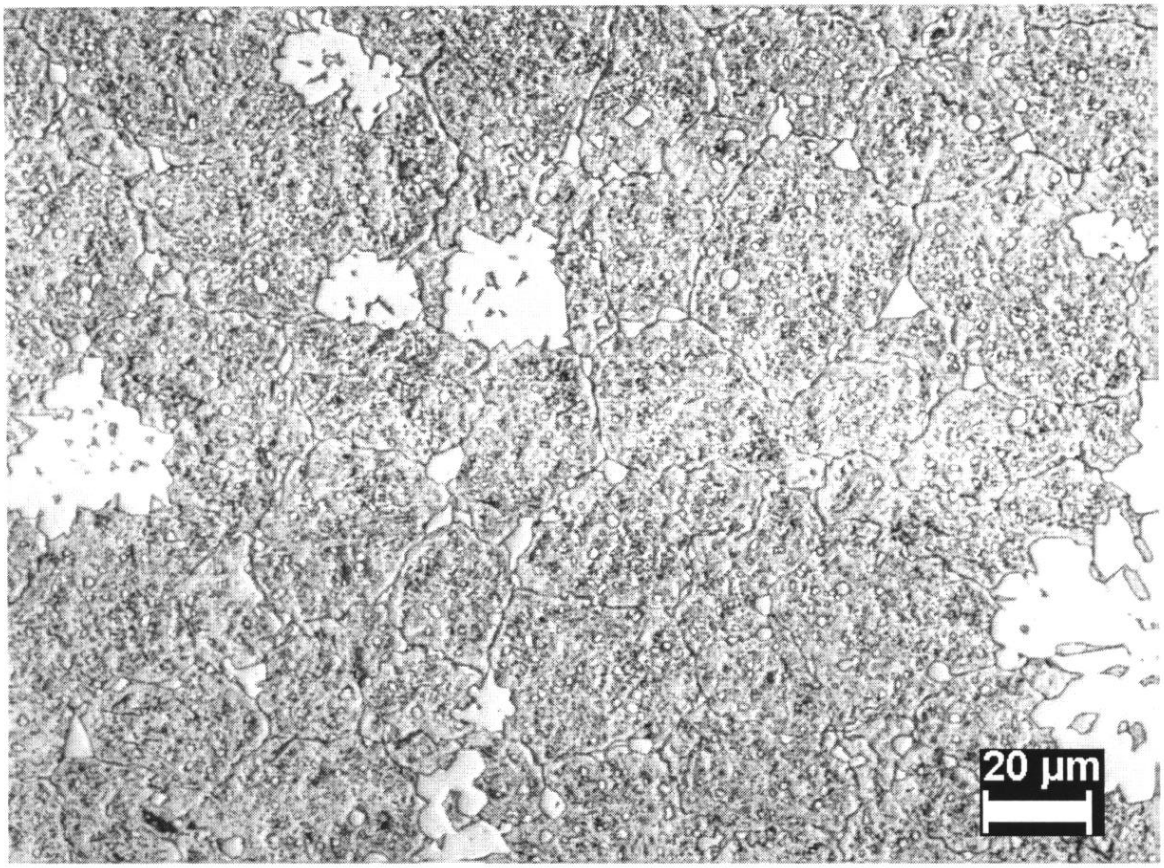

Figure 7: M2 tool steel +6% WC sintered in $N_2$ + 5% $H_2$ at 1250°C for 60 minutes.