

FIELD EXPERIENCE ON A NEW SINTER-HARDENING MATERIAL

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**Presented at PM²TEC 2000
International Conference on Powder Metallurgy & Particulate Materials
MAY 30 – JUNE 3, 2000 New York City**

ABSTRACT

Traditionally, the processing of sinter-hardening materials has been limited to conventional sintering temperatures. Hence, very little sinter-hardening research has been conducted at higher sintering temperatures. However, the superior hardenability of Ancorsteel[®] 737 SH allows for sinter-hardening at temperatures in excess of 1180 °C (2150 °F) without the need for accelerated cooling.

This paper will both present field experience on conventional sinter-hardening processes and investigate the effect of copper, nickel, and graphite additions on the properties of Ancorsteel 737 SH sintered at 1260 °C (2300 °F) utilizing conventional cooling. Particular attention will be paid to dimensional change characteristics, mechanical properties, apparent hardness values, and martensite content in sintered parts.

INTRODUCTION

Previous sinter-hardening work has focused on improving mechanical performance and apparent hardness by controlling microstructure through proper material selection and refining of processing conditions [1-5]. Given the volume of published work on the subject, it can be said that a great deal of sinter-hardening experience exists for processing at conventional sintering temperatures. However, by processing a sinter-hardening material at higher sintering temperatures, this effort represents an initial step toward increasing the capabilities of a recently developed sinter-hardening powder.

It is widely believed that substantial martensitic transformation is unattainable in high temperature sintering furnaces due to the relatively slow cooling rates from the sintering temperature. However, as seen in early development efforts and subsequent processing experience, accelerated cooling often is not required for the evolution of significant percentages of martensite when the new sinter-hardening material is employed. Hence, if

premix compositions are carefully considered, it is felt that high temperature sintering could offer improved mechanical properties without sacrificing hardness.

The ability to high temperature sinter in a production sinter-hardening operation offers a great deal of benefits to the P/M parts manufacturer [6]. Firstly, the rounding of porosity at higher sintering temperature often results in higher mechanical properties and impact energy. More importantly, higher sintering temperatures increase the diffusion rates of copper and nickel and can yield more homogenous microstructures, increased hardenability, and enhanced strength.

FIELD EXPERIENCE

Production experience with the new sinter-hardening material and the sinter-hardening process has continually provided advantages to customers and end-users compared with conventional quenching and tempering. These documented advantages have included lower processing costs, reduced production time, and superior dimensional stability. Dimensional stability includes reduced distortion, tighter concentricity, and improved geometric tolerances.

The capabilities of the new sinter-hardening material have been proven in parts and part families whose design criteria include wear resistance, cleanliness, excellent tensile and yield strengths, good impact energy, and high apparent hardness. Applications for such a material exist in many different industry / product groups. These groups include automotive, lawn and garden, hand / power tools, home appliances, compressors, and industrial applications. Efforts to broaden the market for sinter-hardening materials are undertaken on a daily basis. Such efforts have identified opportunities for sinter-hardening. Table I identifies some opportunities.

Table I: Perceived Part and Part Family Opportunities for Sinter-Hardening

Industry	System	Part / Component Family
Automotive		
	Engine	Timing Sprockets, Valve Seats, Gearing
	Transmission	Gearing, Transfer Case Gearing and Sprockets, Clutch Components
	Chassis	Steering Components, Structural Components, Seat Guides
	Auxiliary Motors	Gearing, Brackets
Lawn and Garden		
	Transaxle Gears	Spur, Bevel
	Structural	Lever Brackets, Blade Adapters, Bushings
Other		
	Various	Gearing, Structural, Valve Guides, Bushings, Wear Plates

Conversion and new application opportunities may increase substantially as enhanced processing routes are explored and production conditions are optimized. After establishing the superiority of a material system under conventional processing conditions, the most obvious method to increase properties is to employ high temperature sintering. Such a study is best done in several steps. This paper represents the first step toward meeting increasingly demanding application requirements.

EXPERIMENTAL PROCEDURE

In an effort to gauge the effect of copper, nickel, and carbon content on the high temperature sintered properties of the new sinter-hardening material, nine premix compositions were chosen for investigation. These compositions are presented below in Table II. A 230 kg (500 lb.) premix was made for each composition. The copper used was ACuPowder 8081 and the graphite was Asbury 3203HSC. In all cases, 0.5 w/o Lonza Acrawax C was added to the premixes.

Table II: Premix Compositions

Copper (w/o)	Nickel (w/o)	Graphite (w/o)
--	--	0.9
0.5	--	0.9
1.0	--	0.9
1.5	--	0.9
2.0	--	0.9
2.0	--	0.7
--	1.0	0.9
--	2.0	0.7
--	2.0	0.9

Green density, green expansion, sintered density, and dimensional change were determined from the average of five compacted transverse rupture strength (TRS) specimens with a nominal size of 6.35 mm x 12.7 mm x 31.75 mm (0.25 inches x 0.5 inches x 1.25 inches). Tensile tests were conducted on machined threaded tensile specimens with a gauge length of 25.4 mm (1.0 inches) and a nominal diameter of 5.08 mm (0.20 inches). Due to the high apparent hardness of the material, tensile specimens were machined by grinding.

All test pieces were pressed to 7.0 g/cm³ and sintered under production conditions in a fully loaded furnace. The furnace used in this study was a CM 12 inch pusher furnace with a standard cooling section. The production sintering cycle for most specimens was as follows:

Sintering Temperature:	1260 °C (2300 °F)
Atmosphere:	50 v/o N ₂ , 50 v/o H ₂
Time in Hot Zone:	42 minutes
Cooling:	Standard

Additional specimens for selected compositions were sintered under normal production conditions:

Sintering Temperature:	1175 °C (2150 °F)
Atmosphere:	95 v/o N ₂ , 5 v/o H ₂
Time in Hot Zone:	50 minutes
Cooling:	Accelerated

The additional specimens served as a baseline for property comparisons. All sintered parts were tempered at 205 °C (400 °F) in air for 1 hour prior to testing or machining.

Apparent hardness measurements were performed on the surface of the specimens using a Rockwell hardness tester. All measurements were conducted on the Rockwell C scale (HRC) for ease of comparison. Transverse rupture strength and dimensional change from die size were measured according to ASTM B 528 and B 610. Tensile testing was performed on a 267 kN (60,000 lbf) Tinius Olsen universal testing machine at a crosshead speed of 0.635 millimeters/minute (0.025 inches/minute). Elongation values were determined by utilizing an extensometer with a range of 0 to 20%. The extensometer was left on until failure.

Metallographic evaluation was performed on sections prepared from machined tensile, TRS, and Charpy impact bars. Photomicrographs showing typical microstructures were taken at an original magnification of 500X following a 2% nital / 4% picral etch. The relative microstructural constituent content was estimated by observing numerous representative fields.

RESULTS AND DISCUSSION

The employment of high temperature sintering was seen to impact all properties of the sinter-hardenable premixes. Particular attention was paid to characteristics such as dimensional change, apparent hardness, impact energy, and tensile properties. Whenever possible, comparisons were made with identical premix compositions processed at conventional sintering temperatures. The reader is referred to reference 5 for a more thorough investigation of properties attained by conventional sinter-hardening processing. Photomicrographs were analyzed in order to quantify the martensite content of the samples.

Apparent Hardness

The data collected for all the new sinter-hardenable material premix compositions are presented in Table III. Not surprisingly, premix compositions were found to have a tremendous effect on the apparent hardness values obtained. More specifically, the data suggested that admixed copper and/or nickel additions were vital for a significant sinter-hardening response under the conditions considered. In contrast, previous work illustrated the ability to achieve high apparent hardnesses at lower sintering temperatures with graphite alone [5]. Given the inherently slow cooling rates in high temperature furnaces, the thorough diffusion of copper and/or nickel was thought to increase the hardenability of the compact and allow for significant martensitic transformation.

Dimensional Change

As expected, the dimensional change of compacts sintered at 1260 °C (2300 °F) was very different from that of compacts sintered at 1175 °C (2150 °F). While nickel premixes were observed to densify more at 1260 °C (2300 °F), the gain in density was coupled with a higher degree of shrinkage from die size. On the other hand, copper containing premixes were seen to densify while exhibiting dimensional changes closer to die size. Figure 1 illustrates the

dimensional change response of premixes with 0.9 w/o admixed graphite at various copper levels.

Although Figure 1 was not intended to accurately predict dimensional change at any given copper level, it effectively conveyed the possibility for a true die size material under the production conditions considered. Under identical testing conditions, a die size material might be achieved at approximately 2.6 w/o copper and 0.9 w/o graphite. Further work to test this hypothesis and to quantify dimensional variability will be conducted in the future.

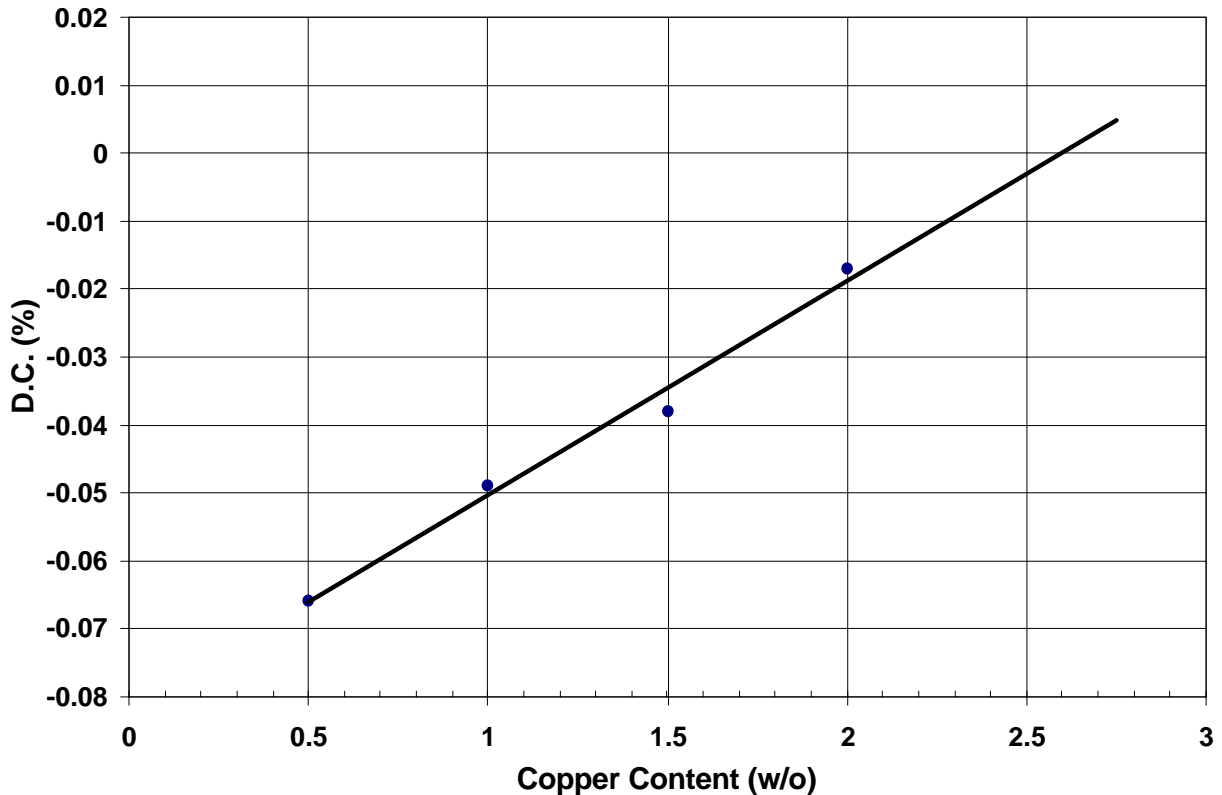


Figure 1: The Effect of Copper Content on Dimensional Change with 0.9 w/o Graphite

Impact Energy

The impact energies of specimens were seen to increase with increasing copper or nickel content. Despite density disparities, no substantial differences were noted between copper premixes and nickel premixes at equivalent carbon levels. The impact energies in Table III represented an improvement over conventionally processed sinter-hardenable premixes [5]. Future work may include a study of tempering temperature and its effect on impact energy.

Tensile Properties

The primary goal of the high temperature sinter-hardening effort was to increase tensile properties in order to meet more stringent application requirements. Specifically, it was hoped that elongations of at least 2.0% could be attained at strength levels greater than or equal to those seen in conventional processes. As evidenced by the data in Table IV, this effort was somewhat successful. Ductility goals were met in specimens whose apparent hardnesses ranged from 13 to 30 HRC.

TABLE III: Properties of Ancorsteel 737 SH Sintered at 1260 °C (HT) and 1175 °C (Conv.)

Copper (w/o)	Nickel (w/o)	Graphite (w/o)	Process	Sintered Density (g/cm ³)	D.C. (%)	TRS (MPa / 10 ³ psi)	Apparent Hardness (HRC)	Impact Energy (J / ft.lbf)
--	--	0.9	HT	7.00	-0.05	1300 / 189	10	16 / 12
--	--	0.9	Conv.	6.98	+0.22	1585 / 230	41	--
0.5	--	0.9	HT	6.98	-0.07	1450 / 210	16	15 / 11
1.0	--	0.9	HT	6.99	-0.05	1725 / 250	27	17 / 12
1.5	--	0.9	HT	6.99	-0.04	1920 / 278	38	19 / 14
2.0	--	0.9	HT	6.99	-0.02	2080 / 300	38	21 / 15
2.0	--	0.9	Conv.	6.93	+0.20	1930 / 280	38	--
2.0	--	0.7	HT	6.95	+0.13	2020 / 293	31	16 / 12
--	1.0	0.9	HT	7.04	-0.22	1775 / 257	28	17 / 13
--	2.0	0.7	HT	7.07	-0.36	2055 / 208	30	19 / 14
--	2.0	0.9	HT	7.08	-0.45	1885 / 270	37	22 / 16
--	2.0	0.9	Conv.	7.02	-0.10	1825 / 265	39	--

NOTE: Properties given for specimens tempered at 205 °C (400 °F) for 1 hour in air

TABLE IV: Tensile Properties for Ancorsteel 737 SH Sintered at 1260 °C (2300 °F)

Copper (w/o)	Nickel (w/o)	Graphite (w/o)	Y.S. (MPa / 10 ³ psi)	UTS (MPa / 10 ³ psi)	Elong. (%)	Apparent Hardness (HRC)
--	--	0.9	580 / 84	765 / 111	2.9	13
0.5	--	0.9	605 / 88	815 / 118	2.4	16
1.0	--	0.9	625 / 91	840 / 122	2.3	23
1.5	--	0.9	760 / 110	1040 / 151	1.5	40
2.0	--	0.9	780 / 113	1045 / 152	1.4	41
2.0	--	0.7	850 / 123	1165 / 169	2.0	31
--	1.0	0.9	660 / 96	915 / 133	2.1	24
--	2.0	0.7	745 / 108	1055 / 153	2.4	30
--	2.0	0.9	695 / 101	1100 / 160	1.7	38

NOTE: Properties given for specimens tempered at 205 °C (400 °F) for 1 hour in air

The most significant results were seen in both the 2 w/o copper – 0.7 w/o graphite and 2 w/o nickel – 0.7 w/o graphite mixes. In addition to achieving high strength levels and adequate hardnesses for many existing applications, elongation values were 2.0 w/o or higher. Furthermore, it was determined that adding 0.7 w/o graphite instead of 0.9 w/o in a 2 w/o copper mix resulted in higher strength and ductility. Similar results were observed in conventionally processed premixes of the new sinter-hardening material containing copper [5]. However, the effect of carbon level in 2 w/o nickel mixes was mixed since yield strength and elongation increased, but ultimate tensile strength decreased. A more extensive investigation is planned to optimize premix compositions for high temperature sintering.

It is extremely important to recognize that a vast array of processing options and properties still remain unexplored. Although a 7.0 g/cm^3 green density was used in this investigation, higher green densities can be achieved without exceeding a compaction pressure of 690 MPa (50 tsi). Hence, sintered densities as high as $7.15 - 7.20 \text{ g/cm}^3$ may be achieved in an effort to increase properties. In addition, both rolling contact fatigue and rotating bending fatigue behaviors are likely to respond favorably to the increase in sintering temperature as pore morphologies change and the degree of sinter increases. Finally, this material system and the P/M industry may benefit immensely from the commercial introduction of accelerated cooling / conveyance systems for high temperature furnaces. Such systems are currently under development to allow for accelerated cooling while minimizing failures from thermal shock.

Metallography

In the interest of brevity, three premixes were chosen to demonstrate the degree of sinter-hardening possible in 0.9 w/o graphite (Figure 2), 2 w/o copper – 0.9 w/o graphite (Figure 3), and 2 w/o nickel – 0.9 w/o graphite (Figure 4) premixes.

Samples prepared from the 0.9 w/o graphite mix was observed to contain only ~1% martensite with the balance being very fine pearlite / bainite. Samples prepared from both the copper and nickel premixes were found to contain high percentage of martensite (~95% in copper and ~90% in nickel) and small amounts of pearlite / bainite (~5% in copper and ~10% in nickel). These values were obtained without considering retained austenite content. In the future, retained austenite content will be quantified in accordance with the procedure presented in reference 7.

CONCLUSIONS

Sinter-hardening has been achieved by high temperature sintering utilizing Ancorsteel 737 SH premixes containing either graphite and copper or graphite and nickel. As a consequence of the slow cooling rates, copper and/or nickel additions were imperative to achieving an acceptable sinter-hardening response. Upon diffusion into the matrix, the copper and nickel served to increase the hardenability of the compact and allowed for more complete martensitic transformation. The effect of graphite in a 2 w/o copper mix was similar to that previously documented at conventional sintering temperatures [5]. Namely, by lowering graphite additions from 0.9 w/o to 0.7 w/o, both strength and ductility were seen to increase. Although a mixed effect was observed in a 2 w/o nickel mix, where higher ductility resulted when graphite additions were reduced from 0.9 w/o to 0.7 w/o.

FUTURE WORK

Although this body of work adds to a vast database of Ancorsteel 737 SH knowledge, further work is warranted in several areas. Future investigations will be conducted on:

- Quantifying RCF and RBF properties of high temperature sintered parts,
- Determining part to part variation in high temperature sintering,
- Coupling ANCORDENSE[®] processing with high temperature sintering in order to reach increasingly stringent application requirements,
- Optimizing premix compositions for use with high temperature sintering,
- Improving green strength for green machining by incorporating new ANCORBOND[®] technologies, and
- Utilizing accelerated cooling and high temperature sintering in an effort to maximize apparent hardness and mechanical properties.

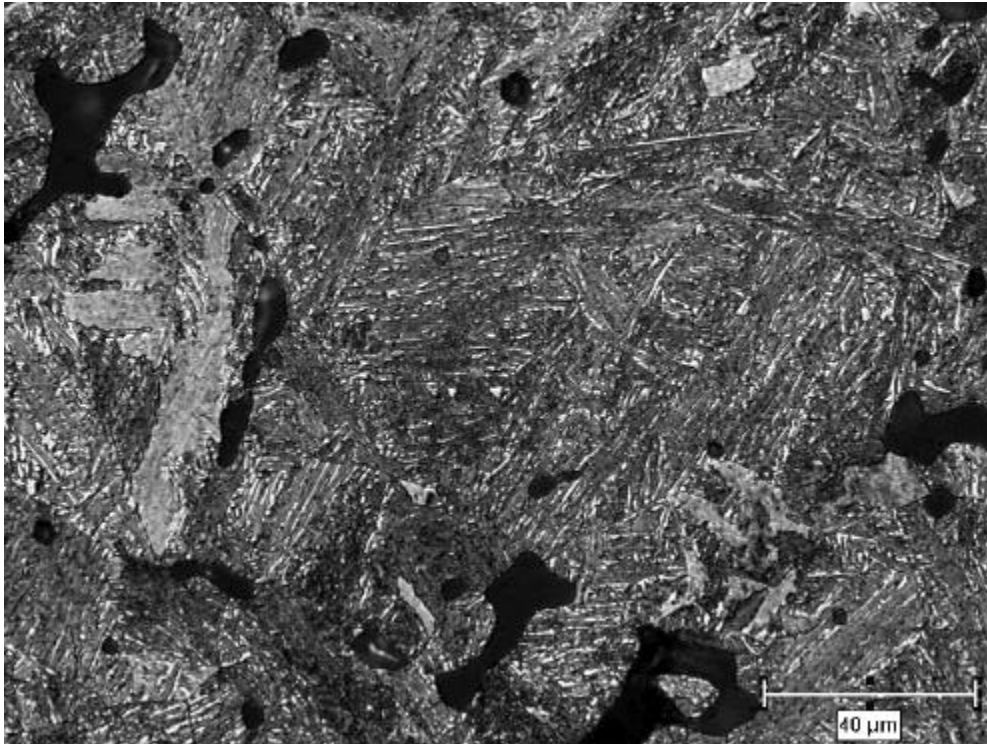


Figure 2: Microstructure of a mix containing 0.9 w/o graphite sintered at 1260 °C (2300 °F). Etched with 2% nital / 4% picral. Original Magnification 500X.

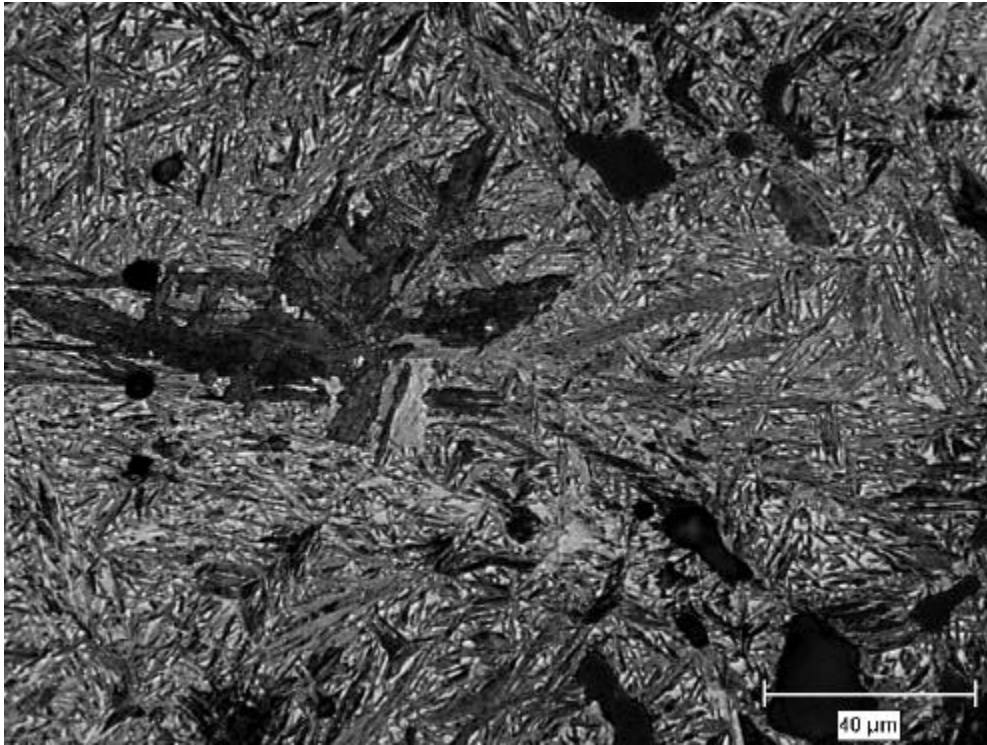


Figure 3: Microstructure of a mix containing 2 w/o copper and 0.9 w/o graphite sintered at 1260 °C (2300 °F). Etched with 2% nital / 4% picral. Original Magnification 500X.

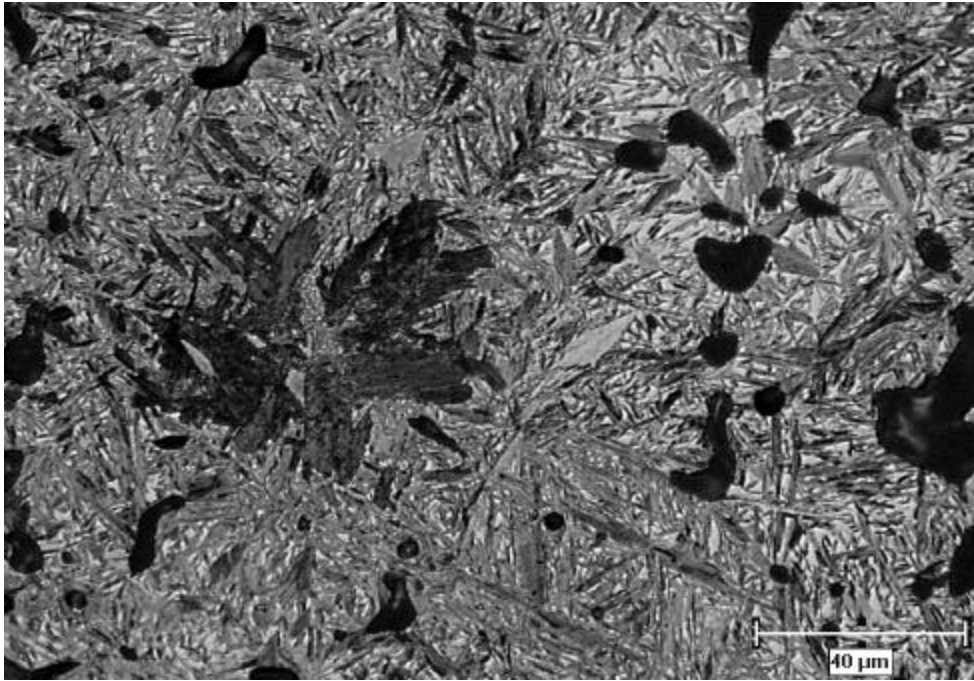


Figure 4: Microstructure of a mix containing 2 w/o nickel and 0.9 w/o graphite sintered at 1260 °C (2300 °F). Etched with 2% nital / 4% picral. Original Magnification 500X.

ACKNOWLEDGEMENTS

The authors would like to express their appreciation to George Fillari, Ronald Fitzpatrick, Gerard Golin, and Steve Kolwicz of Hoeganaes Corporation for all their testing, preparation, and metallography work. In addition, eternal thanks go to John Butterfuss of Alpha Sintered Metals, Inc. for imparting his processing and manufacturing expertise to this effort.

REFERENCES

1. Davala, A.B., Graham, A.H., Causton, R.J., "Effect of Process Conditions Upon Sinter-Hardening Response of FLC-4608 Materials", *Advances in Powder Metallurgy & Particulate Materials–1995*, Vol. 2, Part 14, p.81, Metal Powder Industries Federation, Princeton, NJ.
2. Causton, R.J., James, W.B, Fulmer, J.J., "Performance Characteristics of a New Sinter-Hardening Low-Alloy Steel", *Advances in Powder Metallurgy & Particulate Materials – 1991*, Vol. 5, p.91, Metal Powder Industries Federation, Princeton, NJ.
3. Causton, R.J., Fulmer, J.J., "Sinter-Hardening Low-Alloy Steels", *Advances in Powder Metallurgy & Particulate Materials – 1992*, Vol. 5, p.17, Metal Powder Industries Federation, Princeton, NJ.
4. Davala, A.B., Graham, A.H., Causton, R.J., "Application of High Performance Materials and Processes – Alloy System", *Advances in Powder Metallurgy & Particulate Materials–1998*, Part 13, p.181, Metal Powder Industries Federation, Princeton, NJ.
5. Baran, M.C., Graham, A.H., Davala, A.B., Causton, R.J., Schade, C., "A Superior Sinter-Hardenable Material", *Advances in Powder Metallurgy & Particulate Materials–1999*, Vol. 2, Part 7, p.185, Metal Powder Industries Federation, Princeton, NJ.
6. "Production Sintering Practices," in *ASM Handbook*, Vol. 7, ASM International, Materials Park, OH, 1998, p.474-476.

7. Baran, M.C., Murphy, T.F., "Metallographic Testing to Determine the Influence of Carbon and Copper on the Retained Austenite Content in a Sinter-Hardening Material," *P/M Science & Technology Briefs*, Vol. 1, No. 3, 1999, p. 22-26.