

MICROSTRUCTURAL CHARACTERIZATION OF DIFFUSION-BONDED P/M MATERIALS

L. Tremblay and F. Chagnon
Quebec Metal Powders Limited
1655, Marie-Victorin, Tracy (Quebec) J3R 4R4, Canada

ABSTRACT

P/M parts made from diffusion-bonded powders usually exhibit multiphase microstructures which dictate their mechanical properties. The final microstructure of these materials depends primarily on the carbon content and the sintering temperature, which controls the mass transport mechanisms involved during sintering as well as the cooling rate after sintering.

The effect of carbon content on the microstructure of diffusion-bonded materials (*Fe-0.5Mo-4.0Ni-1.5Cu*) sintered at 1120°C and 1280°C was investigated to optimize tensile, apparent hardness and impact properties. Test specimens containing 0.25, 0.55 and 0.85 % combined carbon were sintered with a close control of the cooling rate to avoid undue variations in both microstructure and mechanical properties. The microstructure and distribution of alloying elements were determined by optical metallography, SEM and electron probe microanalyses. The proportion of microstructural constituents and pore characteristics (porosity, size and shape) were evaluated by image analysis.

This study demonstrated that yield strength, UTS and apparent hardness were significantly improved with carbon content and sintering temperature. The effect of temperature is more pronounced as the carbon content increases. It was demonstrated that an increase of carbon and sintering temperature enhances the diffusion of nickel and thus favors the formation of a more homogeneous microstructure containing a high proportion of hard microconstituents (martensite, bainite) and fine pearlite. A higher percentage of retained austenite and nodular pearlite with increased concentration of carbon was also noticed, but the detrimental effect of these microconstituents were overcome by the formation of such a microstructure and also through releasing stress in martensite by a tempering treatment. The pores in specimens sintered at 1280°C were more spherical. The sphericity was intensified by increasing the carbon content at this high temperature. This, together with a change in microstructure, contributed to strengthen, sinter harden and toughen the diffusion-bonded material, while maintaining satisfactory ductility.

INTRODUCTION

Parts made with diffusion-bonded powders are generally characterized by a heterogeneous microstructure. However, according to several studies, an increase of hard microconstituents such as martensite and bainite in a heterogeneous microstructure strengthens the P/M material [1-5]. Moreover, it is currently recognized that the pore characteristics (pore size, pore shape and distribution) also represent an important contributory factor that might influence tensile strength and ductility [2,5-7]. Irregular and closely spaced pores are greatly detrimental, while smooth pores widely spaced represents the ideal pore configuration to optimize these properties.

As generally reported in the literature, a higher proportion of martensite, a more homogeneous distribution of alloying elements and ideal pore characteristics could be attained by increasing the sintering temperature. This was proven to significantly enhance hardness and tensile strength [2-4,8]. However, there are some discrepancies with regards to the actual effect of carbon content. Indeed, it is likely that the improvement in tensile strength mostly occurs in a certain range of carbon and the optimal concentration of this element strongly depends on the type of treatment applied (as-sintered, heat-treated, tempered, etc.) [3,9-11]. Kravic and Pasquine reported a noticeable improvement in hardness, tensile and fatigue strength of a double pressed and as-sintered 4 % nickel steel material (1121°C) by increasing the combined carbon up to 0.8 % [9]. Khaleghi also observed a similar tendency for quenched and tempered (up to 700°C) diffusion-bonded specimens by raising the carbon content from 0 to 0.5 % [10]. In contrast, only a marginal increase in tensile strength, yield strength and apparent hardness was observed by increasing the carbon content from 0.4 to 0.8 % of as-sintered prealloyed steel specimens at 1121°C (Fe-0.85Mo and Fe-1.82Ni-0.54Mo) [11]. The authors directly attributed this to the formation of a more uniform structure of acicular ferrite and carbides above 0.4 % carbon. Moreover, it was demonstrated that the carbon content could also negatively affect the tensile strength. For instance, Engdahl found that increasing the combined carbon from 0.5 to 1.0 % in as-sintered specimens made of a diffusion-bonded powder contributes to improve the fatigue strength, but also to significantly deteriorate the ultimate tensile strength at 1120°C and 1250°C [3]. This was attributed to a decrease of martensite in favor of retained austenite in the microstructure.

The objective of this study is to evaluate the effect of the final carbon content (0.25, 0.55 and 0.85 %) on tensile, apparent hardness and impact properties of a diffusion-bonded powder (*Fe-0.5Mo-4.0Ni-1.5Cu*) sintered at 1120°C and 1280°C. The cooling rate was closely controlled to prevent undue variation in microstructure. Furthermore, the specimens were tempered to avoid stress in the martensite. Several specimens were also tested as-sintered in order to evaluate the effect of the tempering cycle. Finally, the microstructure and pore characteristics (porosity, shape, size) were examined and related to the mechanical properties. This permitted a better understanding of the formation mechanism for all microstructural features (microconstituents and pores), which can be used to optimize tensile, apparent hardness and impact properties of the diffusion-bonded material.

EXPERIMENTAL PROCEDURE

The test material was an Fe-0.5Mo-4.0Ni-1.5Cu diffusion-bonded powder. This powder was mixed with 0.75 % zinc stearate and various graphite contents (SW 1651) to achieve 0.25, 0.55 and 0.85 % combined carbon. Regular “dog-bone” and unnotched Charpy specimens were pressed to a green density of 7.0 g/cm³. The sintering was carried out in a laboratory vacuum furnace kept under an argon atmosphere at 1120°C and 1280°C for 30 minutes. The lubricant burned off was done at 840°C for 30 minutes in a tube furnace under dissociated ammonia to avoid any contamination of the vacuum furnace. The specimens were cooled at a rate of 0.64°C/s from 950°C to 450°C using a variable pressure system to closely control the cooling rate after sintering [4] and tempered at 190°C for 60 minutes in air.

As well, specimens containing 0.55 and 0.85 % C were sintered in a conventional mesh belt furnace (1120°C, 25 min / D(NH₃)) to verify the effect of the sintering conditions and the tempering cycle (190°C, 60 minutes in air) on tensile properties. Tensile properties, apparent hardness and impact energy were determined according to MPIF standards 10, 43 and 40 respectively. A summary of the test program is given in Table 1.

Table 1. Percentage of Combined Carbon, Sintering and Tempering Temperatures of Trials Conducted in Vacuum and Belt Furnaces

FURNACE	COMBINED CARBON (%)	TEMPERATURE (°C)	
		Sintering	Tempering
Vacuum	0.25	1120	190
Vacuum	0.55	1120	190
Vacuum	0.85	1120	190
Vacuum	0.25	1280	190
Vacuum	0.55	1280	190
Vacuum	0.85	1280	190
Belt	0.55	1120	-
Belt	0.55	1120	190
Belt	0.85	1120	-
Belt	0.85	1120	190

The specimens were also analyzed using an optical microscope and a Scanning Electron Microscope (JEOL JSM-5800) to obtain a general overview of the microstructure after etching in a solution containing 4 % Picral and 2 % Nital.

The pore characteristics (porosity, shape and size) and the percentage of microstructural constituents were measured according to a procedure previously used and described in another study [4]. The pore shape was evaluated by eliminating pores smaller than 5 µm in length since these were mostly round. All of these analyses were performed using a Clemex 640 image analyzer. The distribution and concentration of carbon and alloying elements (Ni, Cu) were

evaluated by using a mapping and step scanning probe microanalyzer (CAMECA SX50). Finally, the retained austenite was determined by performing an X-ray diffraction analysis.

RESULTS AND DISCUSSION

Static Properties and Apparent Hardness

Effect of carbon content

Figure 1 illustrates the effect of carbon content and sintering temperature on UTS and yield strength. In accordance with several earlier investigations [9,10], these properties increase with carbon content and sintering temperature. The UTS and yield strength reached respectively 875 and 620 MPa with a 0.85 % C mix at 1280°C. It is worth noting the more pronounced effect of the carbon content as the sintering temperature is increased from 1120 °C to 1280°C. Therefore, the effect of temperature is much more important at 0.85 % C. The gain in UTS and yield strength by raising the sintering temperature is respectively 250 and 150 MPa at 0.85 % C compared to only 65 and 50 MPa at 0.25 % C. It was also observed that the improvement in yield strength with temperature of a specimen containing 0.85 % C is noticeably lower than that observed for the UTS. Indeed, yield strength rapidly increases from 0.25 to 0.55 % C, but tends to reach a plateau from 0.55 to 0.85 % C at 1280°C.

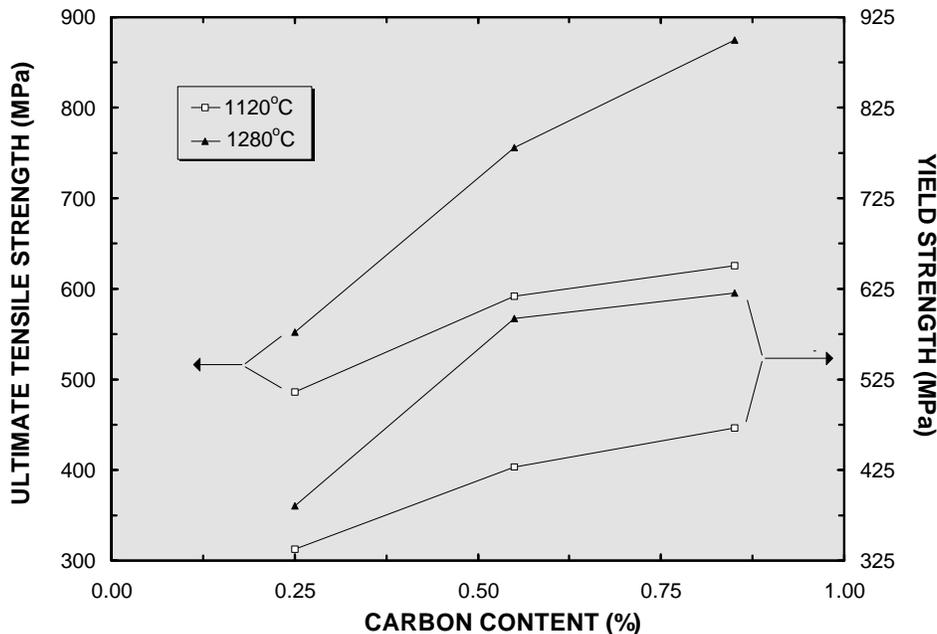


Figure 1. Effect of Carbon Content and Sintering Temperature on UTS and Yield Strength of Tempered Fe-0.5Mo-4.0Ni-1.5Cu Materials.

Figure 2A shows the effect of carbon content and sintering temperature on apparent hardness. This property increases with the carbon content for both sintering temperatures. As observed for strength, the beneficial effect of sintering temperature on apparent hardness is more pronounced at 0.85 % C than at lower carbon contents. A value of 101 HRB (24 HRC) is reached for the 0.85 % C specimens sintered at 1280°C. Improved diffusion of the alloying elements makes this material suitable for sinter hardening applications.

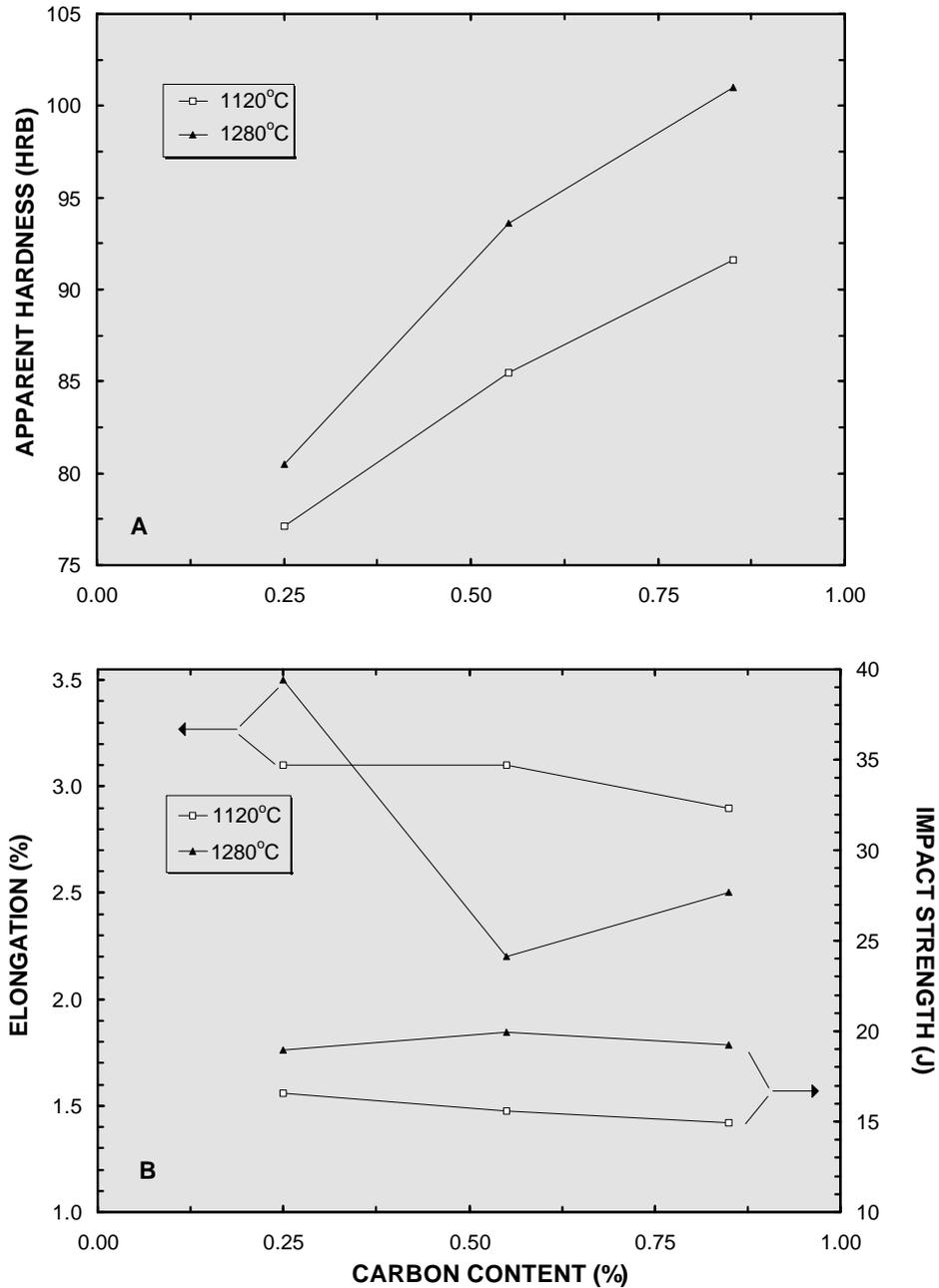


Figure 2. Effect of Carbon Content and Sintering Temperature on Apparent Hardness, Elongation and Impact Properties of Tempered Fe-0.5Mo-4.0Ni-1.5Cu Materials.

On the other hand, it was also observed that increasing the apparent hardness by raising the carbon content also tends to reduce elongation, particularly with high temperature sintering (Figure 2B). The elongation decreases from 3.5 to 2.5 % when increasing the carbon content from 0.25 to 0.85 % at 1280°C, while it only slightly decreased from 3.1 to 2.9 % at 1120°C.

The carbon content was found to have little effect on impact strength (Figure 2B). This property remains practically unchanged as a function of the carbon content at both temperatures. However, impact strength is enhanced by increasing the sintering temperature. This was expected since a more active sintering at high temperature normally favors pore rounding [12].

It is noteworthy that the UTS and apparent hardness reported in this study could be enhanced by using a conventional sintering treatment. As shown in Table 2, these properties are lower than those obtained in a belt furnace kept under an 90N₂-10H₂ atmosphere (1120°C, 25 min) by 100 MPa and 8 HRB respectively. The yield strength is only slightly affected. As previously reported, this difference is probably due to the lower sintering efficiency of the argon atmosphere, which was required for precise control of the cooling rate in the vacuum furnace [4].

Table 2. Tensile Strength and Apparent Hardness of Specimens Containing 0.55 % C, Sintered in Belt and Vacuum Furnaces

FURNACE	PROPERTY		
	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Apparent Hardness (HRB)
Belt Vacuum	695	465	94
	595	430	86

tempered at 190°C for 60 minutes

Effect of the tempering cycle

As shown in Figure 3, the UTS and yield strength are significantly decreased by raising the carbon content from 0.55 to 0.85 % for as-sintered specimens, while these properties increase or remain almost unchanged for tempered specimens. Engdahl also found a large decrease of UTS when raising the carbon content from 0.5 to 1.0 % in as-sintered materials at 1120 °C and 1250°C [3]. Therefore, it can be assumed that the beneficial effect of increasing the carbon content in the range of 0.55 to 0.85 % will only be realized by performing a tempering cycle.

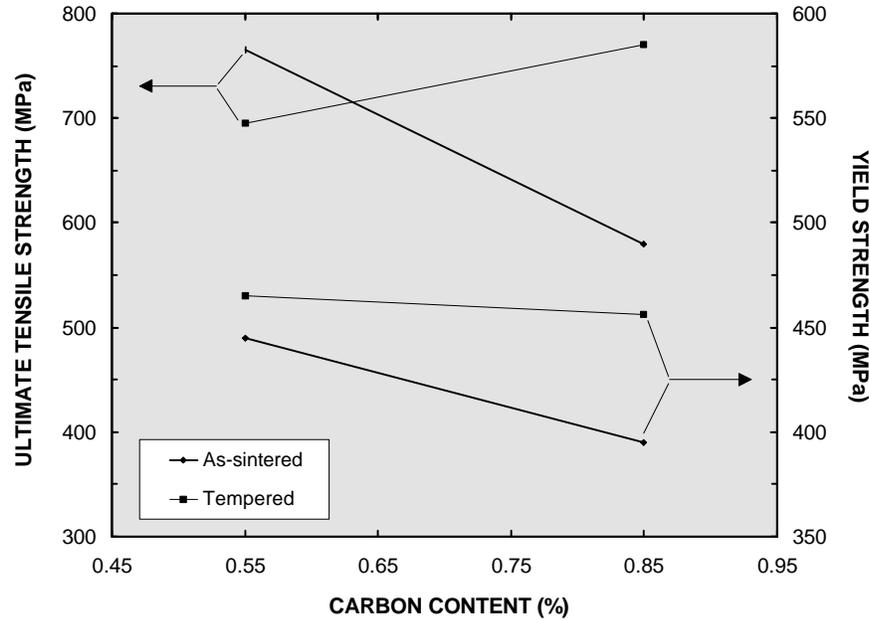


Figure 3. Ultimate Tensile Strength and Yield Strength as a Function of Carbon Content for As-Sintered (1120°C) and Tempered Specimens in a Belt Furnace.

Mechanism of Formation of Microconstituents

Effect of the carbon content

The carbon content and sintering temperature are critical control parameters in attaining the desired microstructure for optimizing tensile properties and apparent hardness. An increase of carbon content from 0.25 to 0.85 % favors the formation of hard phases (martensite, bainite), a finer pearlite structure and a reduction of ferrite at both sintering temperatures (Figures 4A and 4B, Table 3). The Ni-rich δ also decreases, but the total amount of retained austenite in microstructure (Ni-rich regions and finely dispersed through martensite) is still increased by raising the carbon content. These microconstituents are also combined with a larger amount of nodular pearlite even with high temperature sintering. It is worth mentioning that the proportion of hard phases is nevertheless higher at 1280°C relative to that observed at 1120°C and this difference increases with carbon content. Indeed, the effect of temperature is more important at 0.85 % C as shown in Figure 4 and Table 3. An increase of martensite and bainite of around 35 % is obtained by raising the sintering temperature from 1120°C to 1280°C at 0.85 % C compared to only 5 % at 0.25 % C. The resulting pearlite and martensite are also respectively finer and more defined, while the proportion of nickel-rich δ is considerably lower than that obtained at 1120°C (Figures 4D vs. 4B).

The formation of a higher amount of martensite, bainite and finer pearlitic structure as a function of carbon content could be directly related to a higher concentration of carbon in specimens, but also to a more active diffusion of nickel as generally observed with an increase of sintering temperature [4]. Indeed, a part of martensite and bainite is formed at the expense of a certain amount of Ni-rich regions and pearlite from 0.25 to 0.85 % at both temperatures, while the divorced pearlite was transformed into fine pearlite due to a higher concentration of nickel in this

structure (Tables 3 and 4). The martensite is also more defined for the same reason. It is thought that the carbon from C-rich regions can diffuse towards Ni-rich δ regions and might force the nickel to diffuse into surrounding microconstituents (pearlite, bainite, martensite) during the sintering treatment. As given in Table 4, the mean concentration of nickel in Ni-rich δ decreases from 15.0 to 10.0 % by increasing the carbon content from 0.55 to 0.85 % at 1120°C, while it noticeably increases in pearlite and martensite structures. The nickel in γ is limited to around only 4.0 % at 1280°C, the diffusion rate being significantly higher at this temperature. The carbon content in Ni-rich δ reaches about 0.70 %.

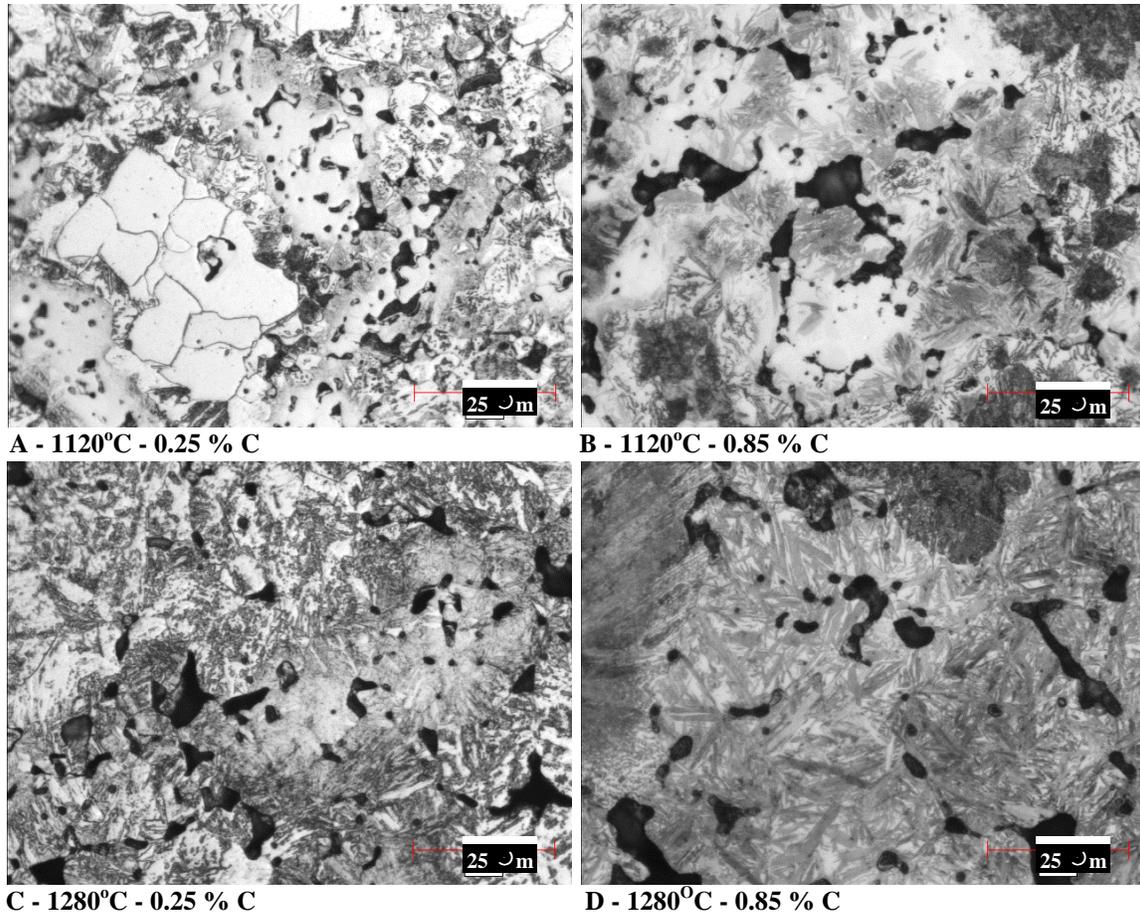


Figure 4. Microstructure of Tempered Fe-0.5Mo-4.0Ni-1.5Cu Material Obtained as a Function of the Carbon Content at 1120°C and 1280°C.

Effect of the tempering cycle

The effect of the tempering cycle on the percentage of retained austenite in specimens containing 0.55 and 0.85 % combined carbon is illustrated in Figure 5. This treatment only slightly affects the total amount of retained austenite in microstructure (Ni-rich region and finely dispersed through martensite) at both carbon contents. However, this treatment likely releases stress in martensite even with specimens containing relatively high carbon contents. As illustrated in Figure 1, UTS and yield strength significantly increase as a function of the carbon content at both

sintering temperatures. The stress in martensite was released by forcing the diffusion of trapped carbon atoms. These carbon atoms were probably combined with iron to form very fine and well-dispersed cementite particles (Fe_3C). These particles can block the dislocation motion and strengthen the material.

Table 3. Percentage of Microstructural Constituents in the Fe-0.5Mo-4.0Ni-1.5Cu Material as a Function of the Carbon Contents at 1120°C and 1280°C

PHASE	PERCENTAGE (%) ⁽¹⁾					
	VACUUM					
	1120°C			1280°C		
	0.25%C	0.55%C	0.85%C	0.25%C	0.55%C	0.85%C
Dark region						
Pore	14	12	11	11	10	10
Nodular Pearlite	<1	11	21	<1	8	15
Dark gray region						
Dark Martensite	5	7	20 ⁽²⁾	10	47 ⁽²⁾	55 ⁽²⁾
White region⁽³⁾						
Retained Austenite ⁽⁴⁾	-	<1	15	-	<1	13
Nickel-rich Austenite ⁽⁵⁾	20	15	9	12	6	2
Nickel-rich Ferrite	25	<1	<1	10	<1	<1
Gray and White region						
Pearlite (fine/divorced)	35 ⁽⁶⁾	53	23	56 ⁽⁶⁾	27	4

(1) average of 15 fields of $1.86 \times 10^5 \mu\text{m}^2$ (2) contains some bainite (3) total retained g measured using an X-ray diffractometer (4) finely dispersed through martensite (5) measured using an image analyzer (6) high proportion of divorced pearlite

Table 4. Carbon and Nickel Content Ranges Detected in Different Microconstituents

PHASE	CONCENTRATION OF ALLOYING ELEMENTS (WT. %)							
	Carbon				Nickel			
	1120°C		1280°C		1120°C		1280°C	
	0.25%C	0.85%C	0.25%C	0.85%C	0.25%C	0.85%C	0.25%C	0.85%C
Martensite ⁽¹⁾	□ 0.35	□ 0.65	□ 0.30	□ 0.90	□ 2.0	□ 4.0	□ 3.0	□ 4.0
Pearlite	0.50-0.75	0.70-0.75	0.50-0.75	0.60-0.80	□ 1.0	□ 2.5	□ 1.5	□ 3.5
Nodular Pearlite ⁽²⁾	0.55-0.75	0.80-1.0	0.50-0.75	0.80-0.85	□ 0.1	□ 0.1	□ 0.1	□ 0.1
Ni-rich Austenite	□ 0.40	□ 0.70	□ 0.50	□ 0.70	□ 15.0	□ 10.0	□ 4.0 ⁽³⁾	□ 4.0 ⁽³⁾

(1) dark/light, contains some bainite (2) may contain traces of ferrite (3) contains finely dispersed martensite

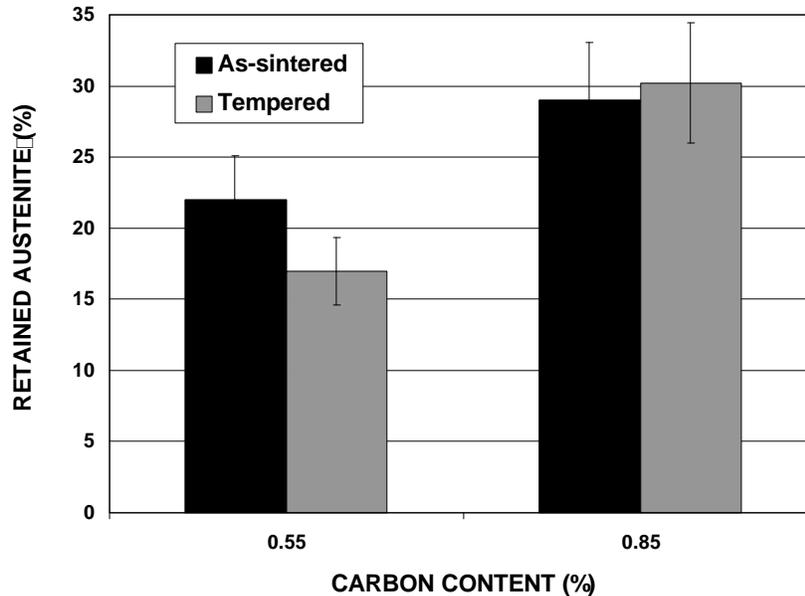


Figure 5. Effect of Tempering Cycle on the Percentage of Retained Austenite (Ni-rich region, finely dispersed through martensite) in Specimens Containing 0.55 and 0.85 % Combined Carbon Sintered at 1120°C in a Belt Furnace.

Considering the mechanical properties illustrated in Figures 1 and 2A, the ideal microstructure to strengthen and sinter harden the diffusion-bonded material should include a high proportion of hard constituents (tempered martensite, bainite) and very fine pearlite with only a few islands of ferrite. This microstructure is obtained by high temperature sintering specimens containing a high carbon content. Note that the detrimental effect of the nodular pearlite and retained austenite by raising the carbon content was probably overcome with the formation of such a microstructure and also by releasing stress in martensite with the tempering treatment

Pore characteristics

The pore characteristics (porosity, pore shape and size) were evaluated for both sintering temperatures (1120°C and 1280°C) on Charpy specimens containing 0.25, 0.55 and 0.85 % combined carbon.

Porosity

Table 5 summarizes the percentage of porosity as a function the carbon content at 1120°C and 1280°C. The porosity increases by raising the carbon content from 0.25 to 0.85 at 1120°C. This is related to the graphite (or added carbon) that may leave voids in the sintered specimen after diffusing. As expected, the porosity decreases with sintering temperature, but remains the same at any carbon content. The higher reduction rate of FeO at 1280°C probably permits a more complete densification of the iron particles during sintering via diffusion processes. Moreover, the densification rate was probably high enough to reduce small voids left by the admixed graphite, even for a relatively high concentration of 0.85 %.

Table 5. Percentage of Porosity Obtained for Tempered Specimens as a Function of the Carbon Content at 1120°C and 1280°C in a Vacuum Furnace

CARBON CONTENT (%)	PERCENTAGE OF POROSITY (%)
Normal temperature (1120°C)	
0.25	12
0.55	12
0.85	14
High temperature (1280°C)	
0.25	11
0.55	11
0.85	11

Pore shape and size

Figures 6A and 6B illustrate the effect of carbon content in the pore shape factor (P.S.F.) and pore size for both sintering temperatures. The pore shape was assessed by considering a pore shape factor (P.S.F.). A factor of one corresponds to a perfect circular pore. For the specimens sintered at 1120°C, the P.S.F. distribution of pores larger than 5 μm remains almost unchanged from 0.25 to 0.85 % C, and 55-58 % of pores are less than 10 μm² for both carbon contents. The formation of these pores should be related to the normal porosity in specimens as well as to the diffusion of graphite and nickel that leaves small voids during sintering. Indeed, a large amount of small open and internal (or trapped) pores were detected in Ni-rich regions (Figures 4A and 4B).

With regards to specimens sintered at 1280°C, the pore size is unchanged whatever the carbon content, but the P.S.F. of pores larger than 5 μm is different (Figures 6C and 6D). As illustrated in Figure 6C, the proportion of round pores significantly increases by raising the carbon content from 0.25 to 0.85 % at this high temperature. This could be related to the formation of small pores following the diffusion of graphite and nickel, which easily become spherical at this temperature [12]. The pores obtained are also rounder and coarser (46 vs. 55-58 %) than those obtained at 1120°C with both carbon contents (Figure 6). Referring to Figures 4, 6 and 7, there is a coalescence of large and small (< 10 μm²) open pores, which results in rounder and larger pores with a surface area between 10 and 150 μm². Moreover, it is possible that the very small enclosed pores in Ni-rich regions could be unstable and coalesce through diffusion processes at this high temperature.

The enhancement of UTS and yield strength observed with specimens containing 0.85 % C sintered at 1280°C is explained by a change in microstructure, together with a reduction of porosity and a larger proportion of round pores. Note that higher toughness results from such pore characteristics, but it has only a minor beneficial effect on ductility. Indeed, the densification and pore rounding did not overcome the detrimental effect of martensite and nodular pearlite in the microstructure.

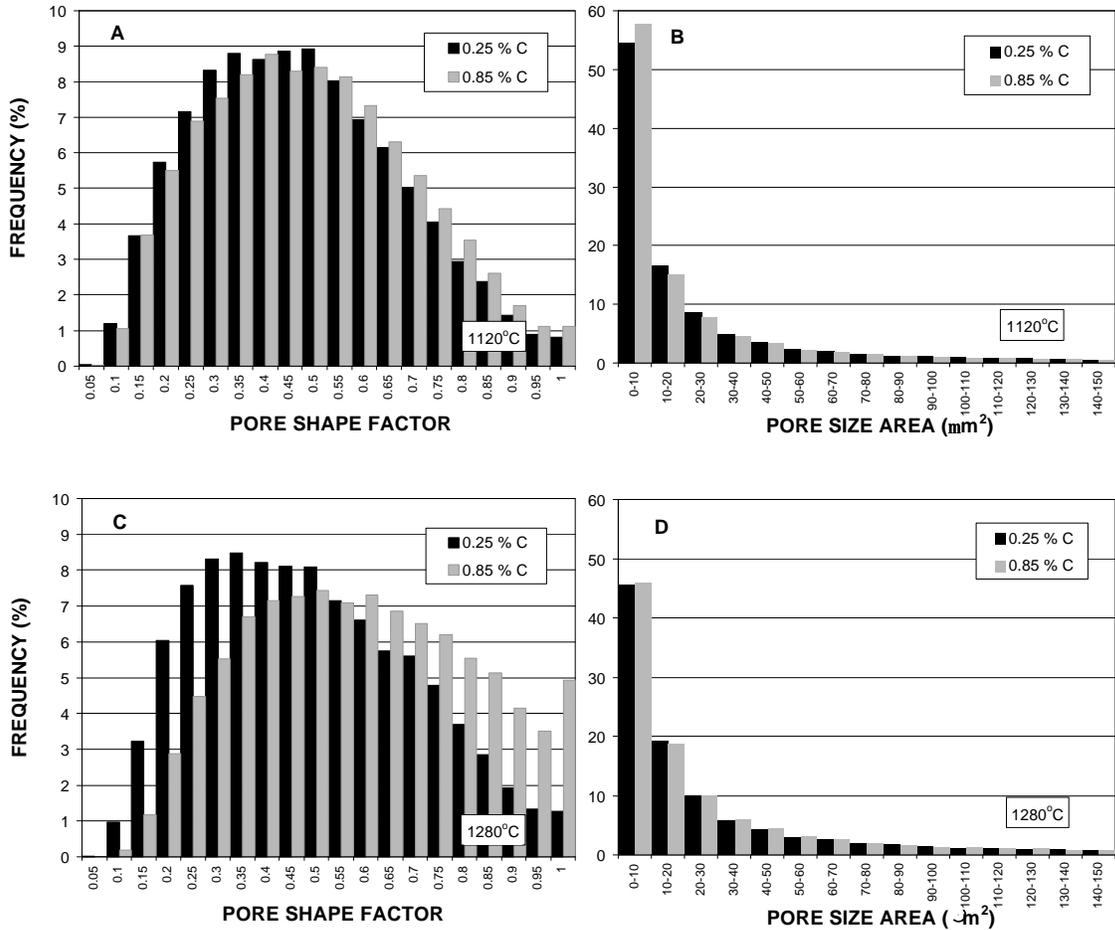


Figure 6. Pore Shape Factor (P.S.F.) and Pore Size Area Obtained as a Function of the Carbon Content at 1120°C and 1280°C.

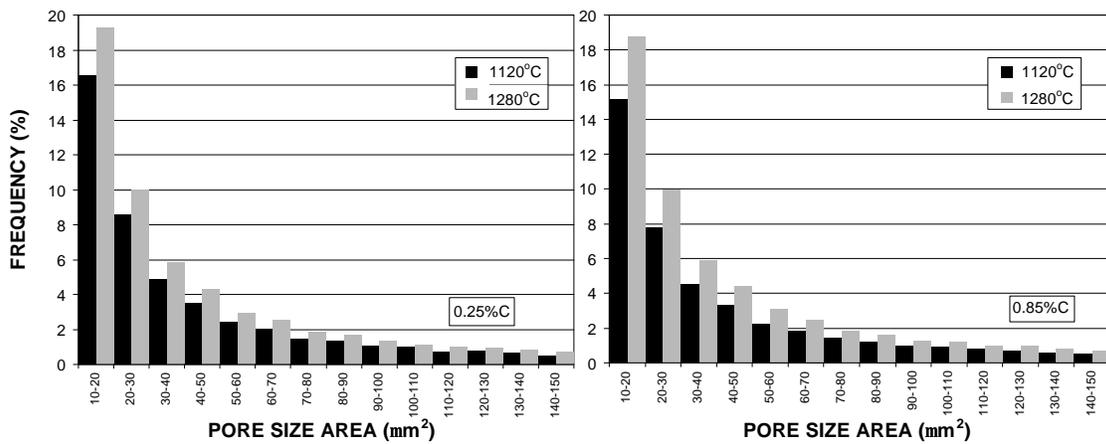


Figure 7. Pore size Area as a Function of the Sintering Temperature with 0.25 and 0.85 % C.

CONCLUSIONS

This study was undertaken to evaluate the effect of carbon content on apparent hardness, tensile and impact properties of specimens made from diffusion-bonded powder sintered at either 1120°C or 1280°C and tempered 60 minutes at 190°C. The following conclusions can be drawn:

- The tensile strength (UTS, yield strength) and apparent hardness of tempered specimens increased with carbon content and sintering temperature. A tempering cycle was required to improve tensile strength at high carbon content.
- As compared to sintering at 1120°C, high temperature sintering (1280°C) significantly increased UTS, yield strength and apparent hardness, particularly for high carbon materials.
- The yield strength increased with carbon content and sintering temperature, but also tended to reach a plateau from 0.55 to 0.85 % C at 1280°C.
- The improvement of UTS, yield strength and apparent hardness with the carbon content and sintering temperature was related to an increase of tempered martensite, bainite and fine pearlite in the microstructure as well as to a change in pore characteristics at 1280°C, more likely an increase of densification and the proportion of round pores as the carbon content increases.
- Increasing the carbon content resulted in a higher percentage of retained austenite and nodular pearlite. However, the detrimental effect of these microconstituents on strength was inhibited by the formation of martensite, bainite and fine pearlite and also through releasing stress in martensite by tempering 60 minutes at 190°C.
- A martensitic rich microstructure, combined with a high amount of nodular pearlite, reduced the elongation of specimens at high carbon content sintered at 1280°C. The increase of densification and pore rounding did not overcome the detrimental effect of these microconstituents on ductility.
- Carbon content had no measurable effect on toughness at either sintering temperature. However, this property is noticeably enhanced with the sintering temperature due to pore rounding and densification during sintering.

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