

Effects of Product and Process Parameters on Dimensional Stability of Sinter Hardened Materials

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Sinter hardening is a cost effective process where PM parts harden during the cooling phase of the sintering cycle. Because of their high inherent strength and hardness in the as-sintered condition, sinter hardened parts are difficult to size or machine through subsequent operations. The control of dimensional change during the processing of these materials is thus of prime importance. A study was conducted to quantify the relative effects of powder properties and that of admixed alloying elements, namely copper and graphite, on the dimensional change of sinter hardening materials. Dilatometric studies were also conducted to characterize the dimensional behavior of these materials during sintering. Results are presented and discussed in terms of their relative influence on the robustness of sinter hardened parts to meet dimensional tolerances.

INTRODUCTION

Sinter hardening is a process in which martensitic transformation occurs when P/M parts are cooled from the sintering temperature. Sinter hardening can offer a significant cost advantage over the quench-and-temper process by reducing the number of process steps to reach the required levels of strength and hardness [1]. However, to achieve high apparent hardness after sintering, base powders used for sinter hardening must possess sufficient hardenability to avoid both the pearlitic and bainitic transformations at cooling rates, usually ranging from 0.4 to 1.5°C/s, of current sintering furnaces. A sufficient addition of graphite in the powder mix is also required to harden the P/M components and copper may also be admixed to further increase hardenability. However, a sizing operation to correct dimensions of the parts after sintering is impossible because as-sintered parts exhibit high apparent hardness. Therefore, the dimensional change must be controlled in a narrow range to avoid part rejection after sintering. Furthermore, for a 2% Cu addition, sinter hardening powders are more sensitive to a variation of the carbon concentration than un-alloyed powders, because of the different diffusion mechanisms and phases produced on cooling [2, 3]. Indeed, a mixture of pearlite/ferrite is present in parts made from un-alloyed steel powders while martensite, bainite and retained austenite will be produced with sinter hardened parts. The tempering treatment can also affect the dimensions of these parts.

The objective of this paper is to review the effect of graphite and copper additions to different sinter hardening powder grades on the dimensional change after sintering and to recommend optimized mix formulations and cooling rates in order to improve the dimensional change control of the components while achieving good mechanical properties.

EXPERIMENTAL PROCEDURE

Three different low alloy steel powders, commonly used for sinter hardening applications, were used for this study. The typical chemical properties of these grades, ATOMET 4601, ATOMET 4701 and ATOMET 4801 (MSP4), are given in Table 1. Mixes were prepared with various graphite additions to reach carbon content after sintering ranging from 0.35 to 0.90% and copper concentrations ranging from 0 to 2%. All mixes contained 0.75% zinc stearate as lubricant. Specimens were pressed to 6.9 g/cm³ and sintered 20-25 minutes at 1120°C in a 90% nitrogen based atmosphere. Sintered properties were determined for cooling rates of respectively 0.4 and 1.5°C/s in the temperature range of 650 to 400°C, before and after tempering 60 minutes at 200°C. The results achieved using laboratory mixes were validated with actual mixes manufactured on a production scale.

Grade	Ni, %	Mo, %	Mn, %	Cr, %
ATOMET 4601	1.8	0.55	0.20	0.05
ATOMET 4701	0.9	1.00	0.45	0.45
ATOMET 4801 (MSP4)	4.0	0.55	0.20	0.05

Table 1. Chemical properties of the three sinter hardening grades.

Dilatometry studies were also carried out with few selected alloys based on ATOMET 4801 to determine their behavior during sintering. These tests were carried out with 15 mm long specimens. As illustrated in Figure 1, the heating rate to the sintering temperature was 10°C/min. with a five minutes isothermal plateau at 625°C to burn off the lubricant. Specimens were held 20 minutes at 1120°C before cooling at a rate of 0.35°C/s from 1120 to 350°C.

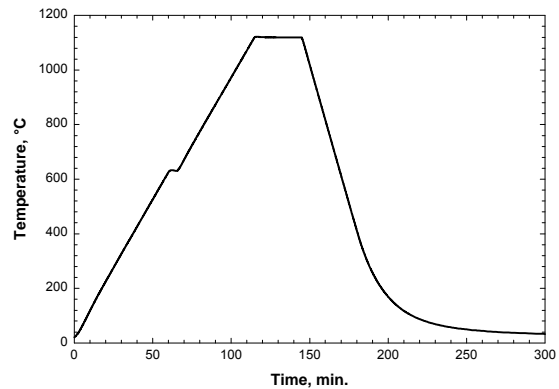


Figure 1. Temperature profiles used for the dilatometry study.

RESULTS AND DISCUSSION

Effect of carbon and copper concentrations and cooling rates on dimensional change

Figure 2 illustrates the evolution of dimensional change from die size of the three materials as a function of combined carbon and copper content, for cooling rates of 0.4 and 1.5°C/s, in the as-sintered condition and after tempering 60 minutes at 200°C. The three materials show similar dimensional behavior as a function of carbon and copper concentrations and cooling rates. In copper free alloys, the dimensional change from die size increases with the carbon concentration while at 1% Cu, dimensional change is less sensitive to carbon variation, particularly for ATOMET 4601 and 4701. At 2% Cu, the dimensional change decreases when increasing the carbon content. It is also worth noting that tempering affects dimensional change, particularly at high carbon and copper contents and at fast cooling rate. This is directly related to the quantity of martensite produced during post-sintering cooling [2].

Dilatometry characterization

Figure 3 illustrates the dilatometry profiles of specimens pressed to 6.9 g/cm³ from mixes based on ATOMET 4801 with carbon and copper contents ranging from respectively 0.35 to 0.90% and 0 to 2%. From room temperature to about 950°C, the various alloys exhibit similar behavior. The sharp increase of the specimen length from room temperature to about 110°C can be related to thermal expansion of lubricant and that of the iron. Subsequently, there is a small plateau at about 150°C, corresponding to the melting of the lubricant and another one at about 425°C corresponding to the lubricant burn off. The rate of increase from 425 to 625°C corresponds to that of the thermal expansion of α -iron, i.e. 0.0014%/°C. After the isothermal plateau at 625°C, the rate of expansion is decreased indicating that some shrinkage occurs in the α phase. Then, an important shrinkage is observed between 700 and 800°C corresponding to the α to γ

phase transformation. This is followed by an increase of the length caused by the thermal expansion of γ -iron and the diffusion of carbon in the iron lattice. From 875 to 950°C, the rate of diffusion of carbon is accelerated, causing an increase of the specimen length.

With copper free specimens, the initiation of shrinkage becomes evident at about 950°C while with copper, a significant growth occurs at about 1080°C which corresponds to the melting temperature of copper. The magnitude of the expansion varies with the amount of copper and carbon. Indeed, the largest expansion values are observed at low carbon content and 2% Cu. This phenomena, known as copper growth, is due to the penetration of liquid copper at the iron grain boundaries which is promoted at low carbon content.

At high carbon content, the dihedral angle of iron grain boundaries is increased which inhibits the penetration of liquid copper [4]. As expected, all specimens show a shrinkage during isothermal sintering at 1120°C varying from 0.16 to 0.24%, depending on the carbon and copper concentrations.

On cooling from the sintering temperature, the various alloys react similarly down to about 650°C with a decrease of the length corresponding to that of the thermal expansion coefficient of γ -iron, 0.0020%/°C. In the range of 650 to 480°C, specimens with 0.38% C at either 0, 1 or 2% Cu exhibit an expansion corresponding to that of γ - α transformation. The temperature at which the transformation starts moves toward lower temperatures as the copper content increases with a decrease of the magnitude of the expansion indicating that the transformation is incomplete as the material hardenability increases. From about 450 to 280°C, there is another phase transformation occurring in specimens with 0.38%C/2% Cu, 0.78%C/1%Cu and 0.98%C/0%Cu. This phase transformation occurs in the temperature range of the transformation of austenite into bainite. Finally, the last transformation starts at about 180°C in alloys with 0.78% and 0.98%C and the three levels of copper. This temperature corresponds to the start of the martensitic transformation and the magnitude of expansion increases with the material hardenability, i.e. as carbon and copper contents increase.

Since the cooling rate measured in the dilatometry studies is similar to that achieved in the sintering furnace, 0.35 vs 0.4°C/s, results from both studies, Figures 2 and 3, can therefore be more easily compared. When analyzed with differential values, results show relatively good correlation considering the different type of

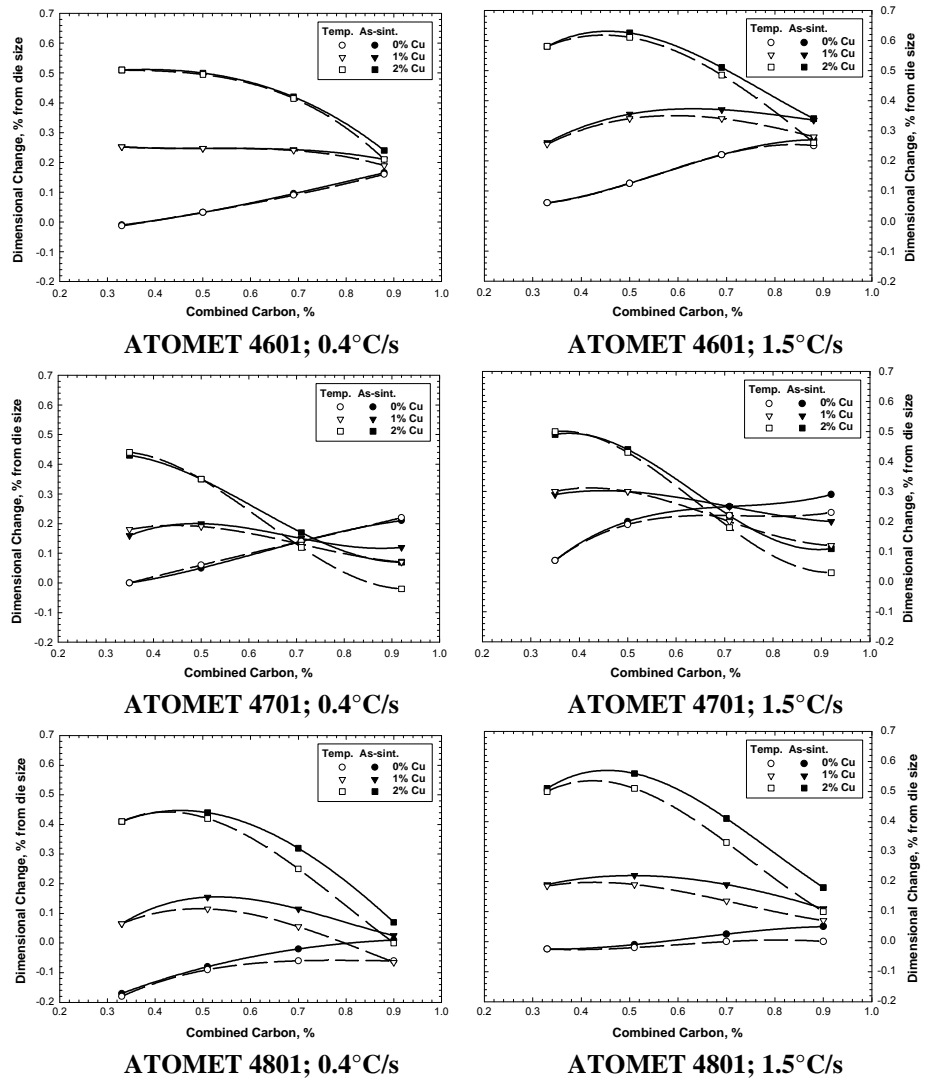


Figure 2. Evolution of dimensional change from die size with combined carbon and copper concentration for cooling rates of 0.4 and 1.5°C/s (density of 6.9 g/cm³).

specimens and furnaces. It is also worth noting that the expansion related to the pearlitic transformation is larger than that of bainite and martensite. This explains the higher sensitivity of dimensional change with carbon content at a copper concentration of 2% because there is no further pearlitic transformation. Also, because hardenability is improved at higher concentrations of carbon and copper, the amount of retained austenite will increase, hence amplifying the difference in dimensional change between low and high carbon concentrations [2].

Optimization of hardness, mechanical properties and dimensional control

In many applications, sinter hardened parts do not only require high hardness but must also meet good mechanical properties while maintaining good dimensional control. As previously mentioned, better dimensional change control is expected at about 1% Cu for the various sinter hardening grades. As illustrated in Figure 4, at that level of copper and for the slow cooling rate, apparent hardness levels off in the range of 35-40 HRC at about 0.7% C with ATOMET 4701 and 4801 while ATOMET 4601, because of its lower hardenability, requires a higher copper concentration to achieve this hardness level. However, when the cooling rate is increased to 1.5°C/s, apparent hardness values of 45 HRC can be reached in the as-sintered condition for the three powders.

Regarding the tensile properties, the three powders react similarly to a change in copper and carbon concentration. As illustrated in Figures 5 and 6, tensile strength increases with combined carbon, reaches a maximum value and tends to decrease at high carbon content. This maximum value moves toward lower carbon content as the copper content and/or the cooling rate increase. This can be related to a larger quantity of martensite in the specimens for these conditions [2]. Also, tempering restores the mechanical properties. Tensile strength values in the range of 950 to 1000 MPa are reached with ATOMET 4701 and 4801 for combined carbon concentrations in the range of 0.5 to 0.6% and 2% Cu for both cooling rates, after tempering for 60 minutes at 200°C. Again, because of its lower hardenability, ATOMET 4601 requires faster cooling rates to reach these strength values. However, if dimensional control is a key parameter tensile strength values in the range of 900-950 MPa can be reached with ATOMET 4701 or 4801 with 1% Cu.

Results achieved with production mixes

For this part of the study, actual mixes produced from ATOMET 4601 were used with graphite and copper contents of respectively 0.9 and 1.2% for mix A and 1.0 and 2% respectively for mix B. Dimensional change, apparent hardness and transverse rupture strength were determined with specimens pressed to 6.8 g/cm³ from a composite sample of the different boxes of each mix. Sintering was performed at 1120°C for

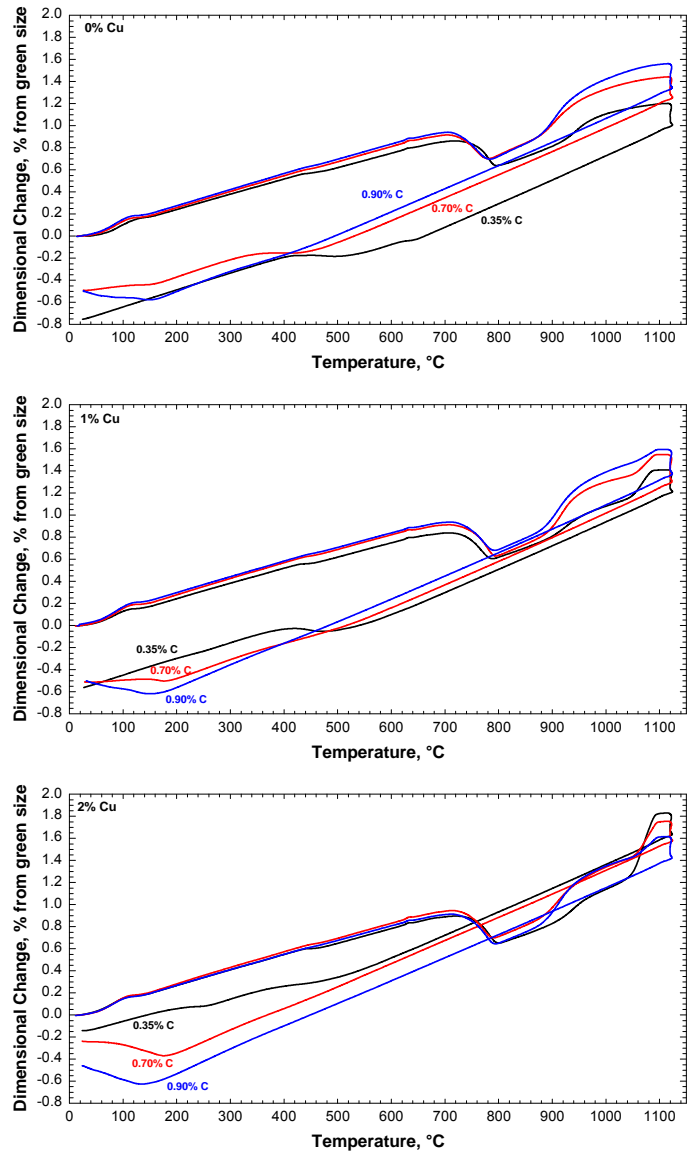


Figure 3. Dilatometry profiles of specimens pressed from ATOMET 4801.

25 minutes in a 90% nitrogen based atmosphere and a cooling rate of 1°C/s in the range of 650 to 400°C. Sintered properties were determined after tempering for 60 minutes at 200°C.

Table 2 summarizes the dimensional change from a standard, apparent hardness and transverse rupture strength results achieved with mixes A and B. Lowering the copper content from 2 to 1.2% reduces the standard deviation of the dimensional change by a factor of about 1.5, from ±0.022 to ±0.015% while the apparent hardness is only reduced by 1.9 HRC and the transverse rupture strength is slightly improved due to the lower carbon content.

CONCLUSIONS

The type and quantity of phases produced when sinter hardened P/M parts are cooled from the sintering temperature significantly affect their sintered properties. A good understanding of the effect of each phase on the dimensional change and on the sintered properties is required to optimize both process control and final properties of sinter hardened parts. In summary:

- The diffusion of carbon from the admixed graphite causes specimen to grow during heating to the sintering temperature. The magnitude of the growth increases with the carbon content.
- Admixed copper favors growth of the specimens when its melting temperature is reached. The magnitude of the growth is function of the copper and carbon concentrations. Larger growth values are observed with higher copper content and low carbon concentration because carbon inhibits copper penetration at the grain boundaries.
- For carbon content above 0.6% with the presence of copper and the use of fast post-sintering cooling rate, the pearlitic transformation is inhibited with materials having high hardenability. The dimensional change of the specimens is then affected by the amount of bainite, martensite and retained austenite produced on cooling.
- Mix formulations with about 1% Cu and enough graphite to reach 0.6 to 0.7 C% after sintering are recommended to improve dimensional change robustness while maintaining good mechanical properties. Faster cooling rates can be used to further increase apparent hardness without penalizing dimensional change stability.

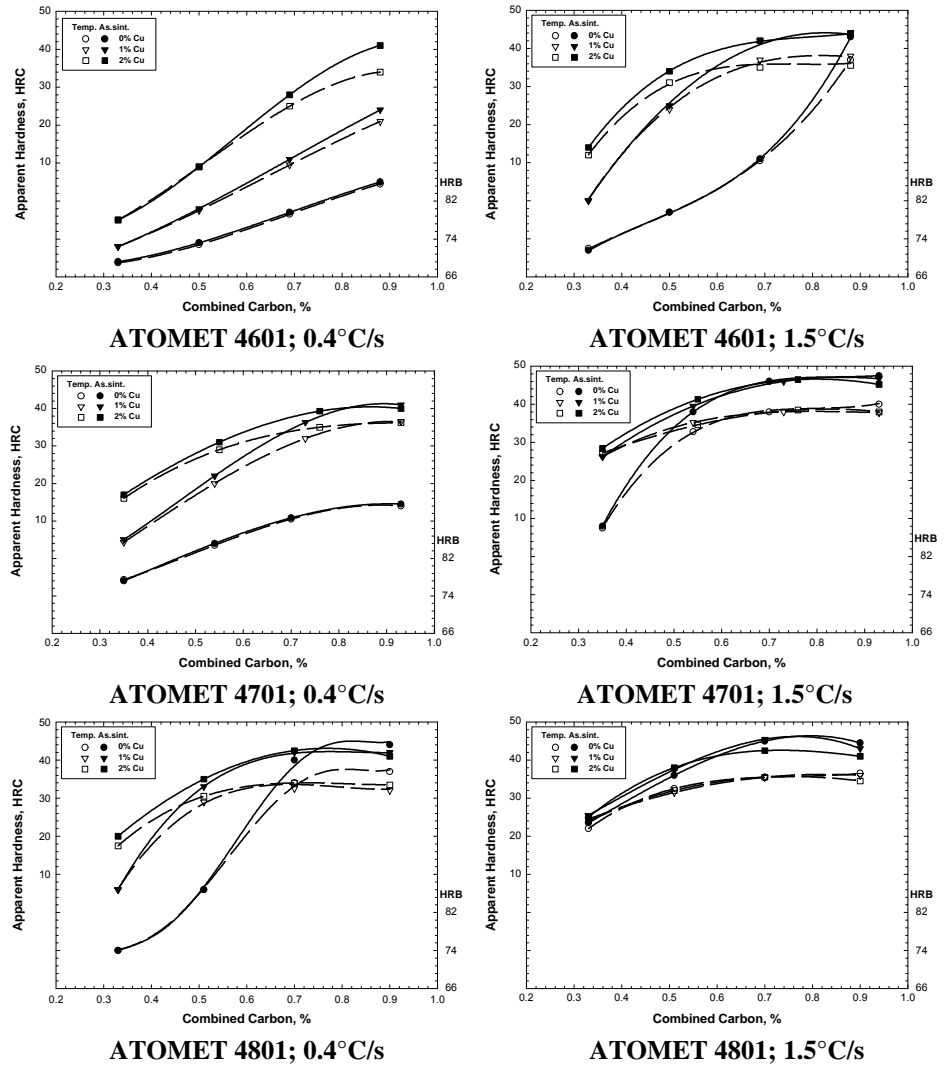


Figure 4. Evolution of apparent hardness with combined carbon and copper concentration for cooling rates of 0.4 and 1.5°C/s (density of 6.9 g/cm³).

- The reduction of the copper content from 2 to 1.2% decreases the dimensional change variation by a factor of 1.5 with powder mixes manufactured on a production scale.

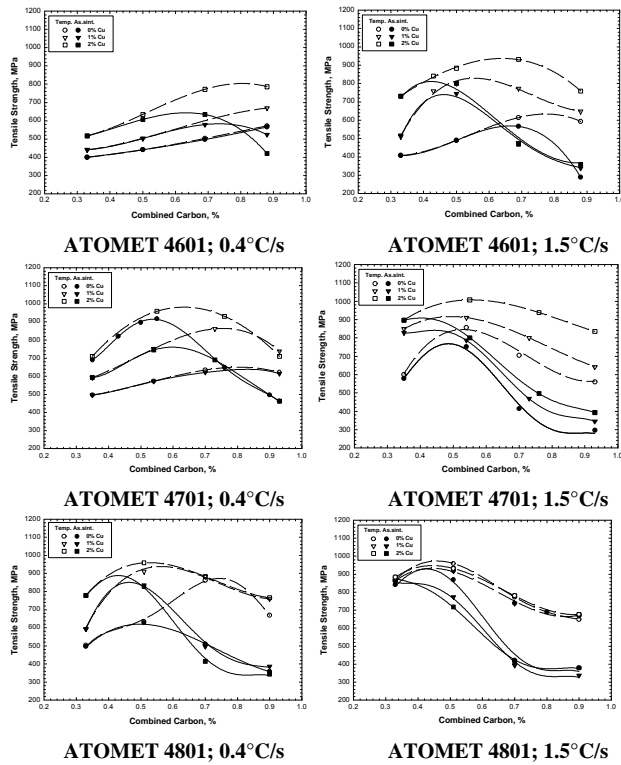


Figure 5. Evolution of tensile strength with carbon and copper contents (green density of 6.9

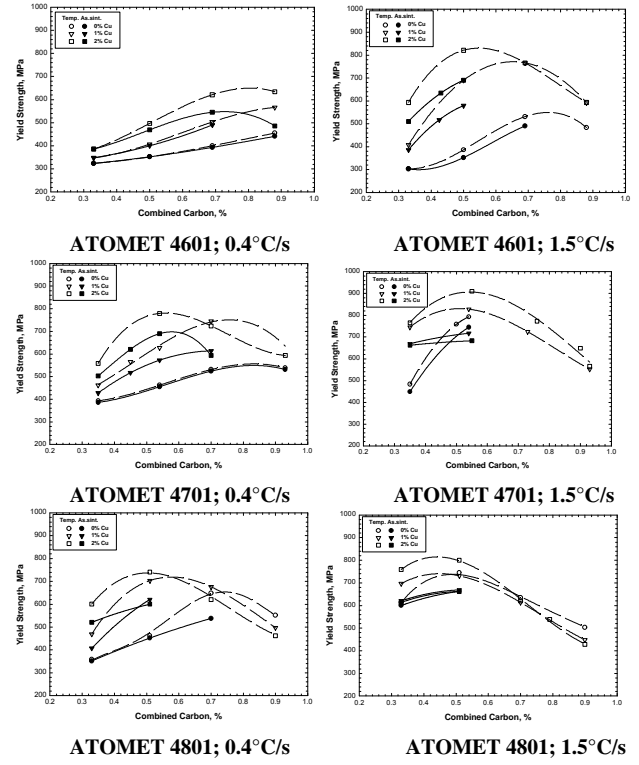


Figure 6. Evolution of yield strength with carbon and copper contents (green density of 6.9 g/cm³).

Id.	Number of lots, n	Dimensional change from standard, %	Apparent hardness, HRC	Transverse rupture strength, MPa
A	18	0.007±0.015	30.7±1.3	1417±74
B	38	0.010±0.022	32.6±0.8	1367±78

Table 2

Dimensional change, apparent hardness and transverse rupture strength of specimens pressed from mixes A and B

REFERENCES

1. E. Iliia, S. Krise, R. Frawley, F. Chagnon and J.A. Danaher; “Cost Reduction Processes for Transfer Case Sprockets. Part 2: Processing Variables”, SAE 2000 World Congress, SAE International, Warrendale, PA, paper 2000-01-1000.
2. F. Chagnon and M. Gagné; “Effect of Graphite and Copper Concentration and Post Sintering Cooling Rate on Properties of Sinter Hardened Materials”; MPIF, Princeton, 2000, pp.13-37 to 13-47.
3. F. Chagnon and M. Gagné; “Dimensional Control of Sinter Hardened P/M Components”; paper presented at the 2001 International Conference on Powder Metallurgy & Particulate Materials, New Orleans, May 2001.
4. Y. Trudel and R. Angers; “Comparative Study of Fe-Cu-C Alloys Made from Mixed and Prealloyed Powders”, Modern Development in Powder Metallurgy, MPIF, Princeton, 1973, pp. 305-322.