

Effect of Sintering Temperature, Heat Treatment and Tempering on Hardness of Sintered Hardened Grade Steels (SH737-2Cu-0.9C)

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Abstract

The present study examines the change in hardness of sintered hardened steel (SH737-2Cu-0.9C) sintered at different temperatures, heat treated by various methods and then tempered at different temperatures. The samples were transient liquid phase sintered at 1120 °C, 1180 °C and 1250 °C respectively. The sintered samples were characterized then for density and densification parameter. The samples were austenitized at 900 °C and cooled by four different methods viz. furnace cooling (annealing), air cooling (normalizing), oil quenching, and brine quenching. The samples were then tested for their hardness using Vickers's hardness at 10 kgf load. The trend of hardness observed was found minimum for air cooled and maximum for brine quenched. In case of sample sintered at 1250 °C, relatively higher hardness was observed. The oil and brine quenched samples were then tempered at 200 °C, 400 °C, 600 °C and 700 °C. The hardness pattern observed typically showed secondary hardness taking place (due to presence of Mn and Mo) and reaching the maximum around 600 °C.

Keywords: Sintered SH737-Cu-C steel, heat treatment, transient liquid phase sintering, quenching, tempering, secondary hardness

1. Introduction

In recent years, powder metallurgical (P/M) stainless steel components are increasingly being utilized for automotive and structural applications.^[1] As compared to conventional casting techniques, P/M processing offers advantages such as lower processing temperature, near-net shaping, high final density, greater material utilization (> 95%) and a more refined microstructure that provides superior material properties.^[2] In addition P/M products have greater microstructural homogeneity. The significant advances in powder production technology, new alloy design with novel properties, compaction and sintering furnace technologies boost up the growth of powder metallurgy.

The main thrust towards higher performance in powder metallurgy alloys has been achieved by introducing alloying additions such as Mo, Ni, Mn, and Cu. Most of the alloying additions enhance the strength through solid solution hardening during

sintering. In addition, these alloying elements also enhance the hardenability by shifting the continuous cooling transformation curve to the right. Subsequent heat treatment results in enhancing the toughness of ferrous alloys. In addition, alloying can improve oxidation or corrosion resistance. Some applications rely on alloying to secure special magnetic properties or high temperature strength. Table 1 qualitatively shows that the alloys that are efficient in improving hardening tend to reduce the compressibility and high affinity for oxygen.

The alloying methods used for the production of ferrous P/M parts are divided into three groups: admixture of elements to a plain iron powder, diffusion bonded or partially prealloyed powders and completely prealloyed powders. Elementally admixed materials suffer heavily from segregation problems. Prealloyed iron powder though effective against segregation, significantly decreases the compressibility of the powders. In conventional powder metallurgy processing, "diffusion alloyed"

powders have typically been used. This process involves heat treatment of iron powder and alloying elements in a reducing atmosphere, allowing partial diffusion and metallurgical bond formation prior to pressing and sintering. Due to partial diffusion, these powders have higher compressibility and fewer tendencies for small alloying elements to agglomerate, hence better homogeneity. The influence of chemical and microstructural homogeneity on the mechanical properties of sintered material has been studied by a number of authors.^[3-5] A common alloying metal in powder metallurgy is copper, which is not sensitive to oxidation and causes sufficient increase in strength. Several sintered components are made for automotive applications by mixing copper and carbon with prealloyed iron. This material is sintered with a transient liquid phase, when copper content is less than 8%.^[6-9] Formation of secondary pores at the site of original Cu particles is an inevitable consequence of transient liquid phase sintering.

This study focuses on the investigation of one such alloy, sintered hardened grade steel SH737 (designated), which has a nominal composition of Fe-1.25 Mo-1.4 Ni-0.42 Mn (wt%). The composition has been tailored with a view to alter the CCT curve characteristics in such a manner that during post sintering cooling, the sintered compact undergoes a transformation in the bainitic/martensitic region. This grade of powder has also

been referred to as a sinter-hardening grade. As the name suggests, sinter-hardening achieves sinter and hardening in a single step.

2. Experimental Procedure

For the present investigation, a partially prealloyed powder mixture (Fe, 1.4 wt% Ni, 1.25 wt% Mo, 0.42 wt% Mn, 2 wt% Cu, and 0.9 wt% graphite), produced using a proprietary process developed by Hoeganaes Corp, was used as a starting material.^[10,11] The as-received powder was characterized for its flow behavior as well as apparent and tap density using set MPIF standards. The results are shown in Tables 2 and 3 and in Figure 1.

The received powder was compacted at 600 MPa in a 50 ton uniaxial hydraulic press (APEX Construction Ltd, UK). To minimize friction, the compaction was carried out using zinc stearate as a die wall lubricant. The density of compacted specimens was between 6.99 and 7.02 g/cm³. The powder contained 0.75 wt. % acrawax, which was added to the powder to facilitate its compaction during sintering. The sintering response on densification and microstructures was evaluated on cylindrical pel-

Table 1. Qualitative ranking of alloying elements in prealloyed materials

	Hardenability factor	Effect on compressibility	Affinity for Oxygen
Higher	Manganese	Copper	Manganese
	Chromium	Nickel	Chromium
	Molybdenum	Chromium	Nickel
	Copper	Manganese	Molybdenum
	Nickel	Molybdenum	Copper
Lower			

Table 2. Composition of the powder

Elements	Fe	Mo	Mn	Ni	Cu	C
% (by wt)	94.03	1.25	1.4	0.42	2	0.9

Table 3. Characteristics of experimental powder

Source	D ₁₀ (µm)	D ₅₀ (µm)	D ₉₀ (µm)	Mode size, (µm)	Width of distribution (µm)	Apparent Density, (g/cm ³)	Tap Density, (g/cm ³)	Flow Time, (s/50g)
Hoeganaes	32	58	88	75	0.47	3.62	3.98	20

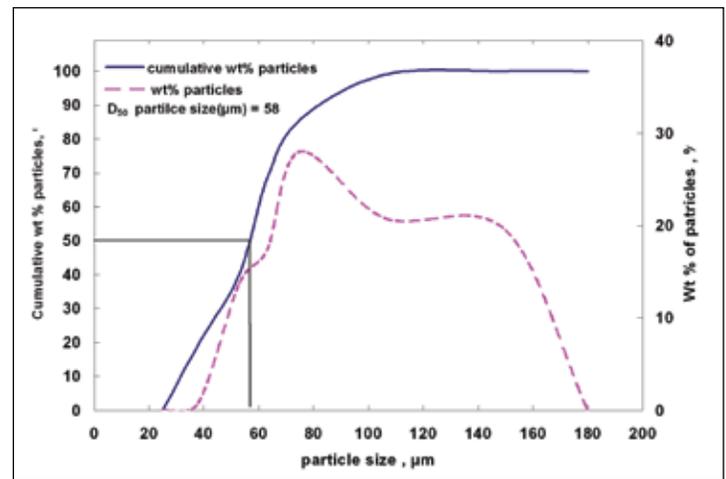


Figure 1. Weight percent of the powder particles vs. particle size

lets (16 mm diameter and 6 mm height). The green compacts were dewaxed in a tubular silicon carbide (SiC) furnace under a 80% N₂-20% H₂ atmosphere. The lubricant was removed from the green samples using a heat treatment at 850 °C for 30 min. To prevent cracking of green compacts by thermal shock, the compacts were slowly heated at 5 °C/min. They were then sintered at three different temperatures, i.e. 1120 °C,

1180 °C and 1250 °C respectively for 30 min in a tube furnace with a SiC heating element. The thermal profiles for the sintering are shown in Figure 2. All the sintering was carried out under a 80% N₂-20% H₂ atmosphere.

The sintered density was obtained by dimensional measurements. The densification parameter was calculated to determine the amount of densification occurred during sintering. It is expressed as:

$$\text{Densification parameter} = \frac{(\text{sintered density} - \text{green density})}{(\text{theoretical density} - \text{green density})} \quad (1)$$

The sintered samples were heat treated at 900 °C for one hour and subsequently cooled by four different methods: furnace cooling (annealing), air cooling (normalizing), oil quenching and brine quenching. The samples were polished to mirror finish and ultrasonically cleaned in acetone, followed by etching in 3% Nital. Bulk hardness of the samples was measured by LECO V - 100 - C1 Vickers hardness tester at 10 kgf load using a pyramidal shaped diamond indenter. The load was applied for 10 seconds. The recorded hardness values are the averages of five readings taken at random spots throughout the sample.

The oil and brine quenched samples sintered at different temperatures were tempered at four different temperatures (200 °C, 400 °C, 600 °C and 700 °C) for 2 hours. The microstructural analysis of the samples was carried out through optical microscopy, and then bulk hardness was measured using Vickers hardness tester at 10 kgf load.

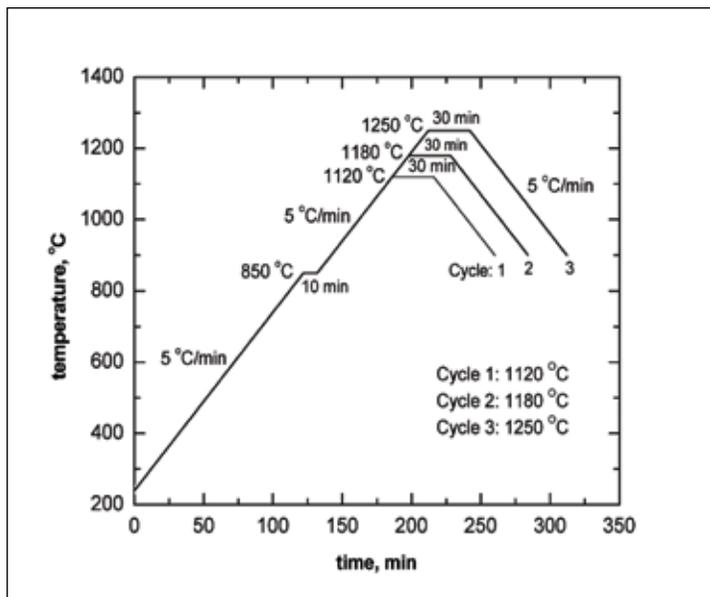


Figure 2. Sintering temperature profiles for SH737-2Cu-0.9C

3. Results and Discussion

Figure 3 shows the effect of sintering temperature on the densification response of SH737-2Cu-0.9C alloys. It is quite evident from the figure that the sintered density improves (~0.5%) with increased in sintering temperature from 1120 °C to 1250 °C. To take into account the effect of composition, all the sintered densities were normalized with respect to the respective theoretical densities. The sintered sample contains nearly 11-12% porosity. It can be inferred that the temperature has marginal effect on the densification of these alloys.

In order to account for the effect of green density on the sintered density, the densification response was qualified in terms of densification parameters. Variation of densification parameter with temperature is shown in Figure 4. The densification parameter is negative for the sintering temperatures 1120 °C and 1180 °C, thus confirming compact swelling. The

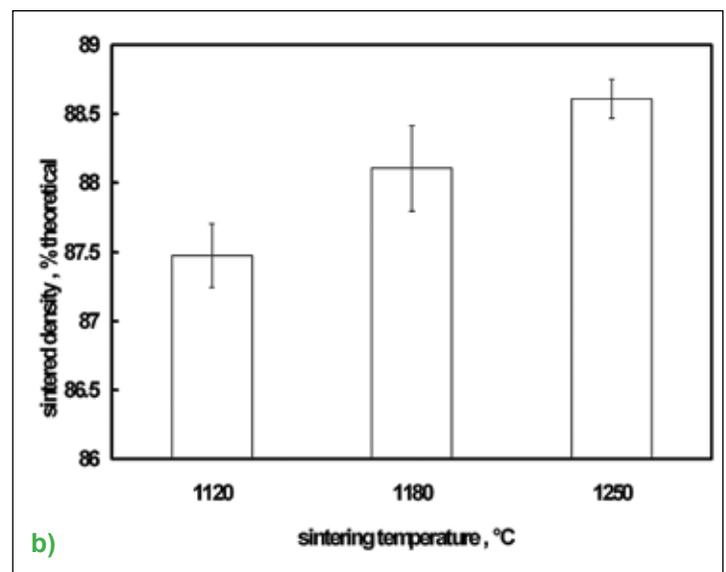
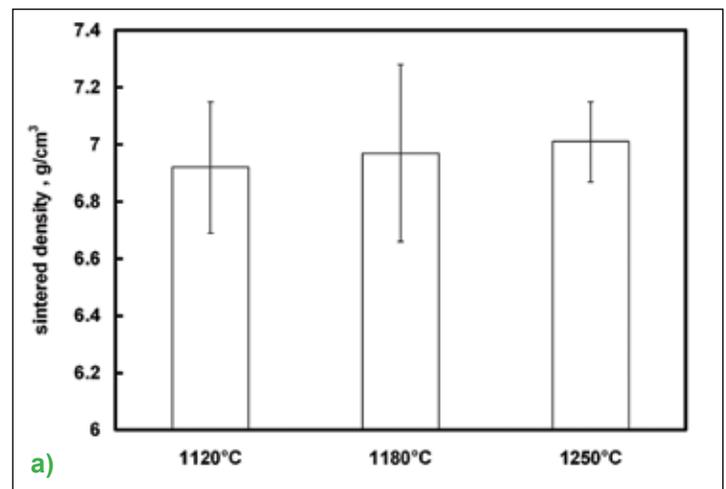


Figure 3. Effect of sintering temperature on densification: a) density b) percent theoretical density

parameter is positive for 1250 °C, thus exhibiting marginal increase in densification parameter with sintering temperature.

Table 4 shows that furnace cooling leads to the lowest hardness regardless of sintering temperature. The trend of hardness is as follows:

Furnace cooled < Air cooled < Oil quenched < Brine quenched

This can be attributed to the formation of martensite and bainite in the case of higher rate of cooling (as in oil and brine quenching).

Figure 5 shows the effect of sintering temperature on the hardness of SH737-2Cu-0.9C alloy samples cooled by various methods. It is evident from the graph that bulk hardness improves with increasing sintering temperature from 1120 °C to 1250 °C, which can be attributed to the sintered density, because there was a marginal densification achieved with increasing sintering temperature. Higher densification implies lower porosity, hence better heat transfer leading to high hardness. A higher sintering temperature further enhances the microstructural

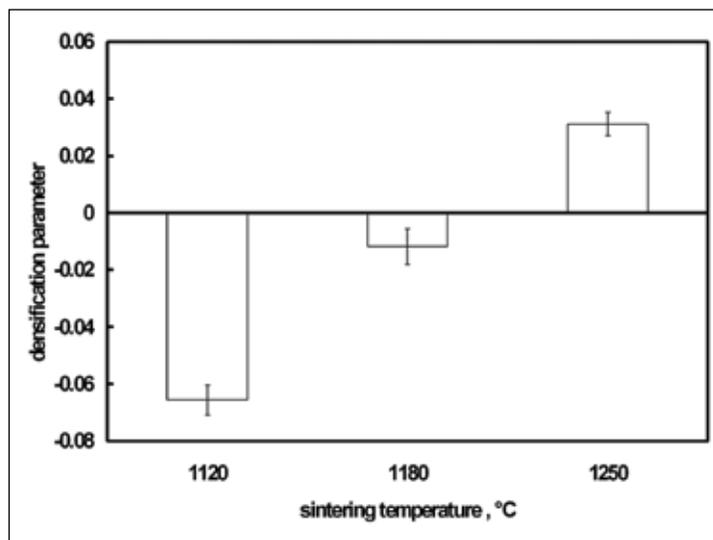


Figure 4. Effect of sintering temperature on the densification response

Table 4. Bulk hardness values of SH737-2Cu-0.9C sintered at three different temperatures (1120 °C, 1180 °C and 1250 °C) and then cooled by different methods

Cooling method	Bulk Hardness Values (HV ₁₀)		
	Sintering Temperature (°C)		
	1120	1180	1250
Furnace Cooled	118±6.0	118±3.1	121±4.6
Air Cooled	148±4.6	147±2.9	156±3.8
Oil Quenched	166±2.5	166±9.5	171±3.7
Brine Quenched	161±7.7	183±3.8	185±6.4

homogeneity due to inter-diffusion of alloying elements. This contributes to the enhancement of the bulk hardness, which also increases with the formation of bainite at higher sintering temperature due to comparatively faster cooling.

Figure 6 and 7 show the hardness pattern of tempered oil and brine quenched samples. The hardness increases up to 600 °C, attains a maximum, and then decreases, which is due to secondary hardening taking place in the quenched samples. Secondary hardening occurs due to presence of carbide formers like Mn and Mo above 500 °C. These elements have high diffusivity to nucleate and form a fine dispersion of alloy carbides producing secondary hardening. Secondary hardening is a process similar to age hardening, where coarse cementite particles are replaced by a new and much finer dispersion of Mo₂C and Mn₂C stable carbides. The critical dispersion causes a peak in the hardness as the carbide particles slowly coarsen, causing their strengthening ability to decrease, leading to an overall decrease in hardness.

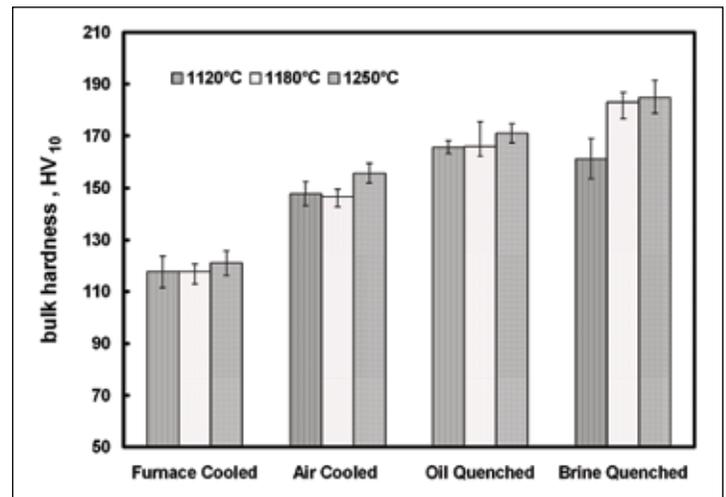


Figure 5. Bulk hardness of SH737-2Cu-0.9C samples sintered at different temperatures and cooled by different methods

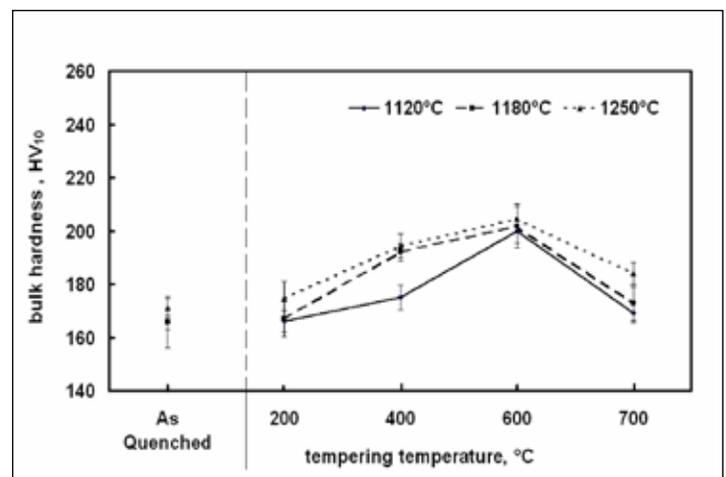


Figure 6. Effect of tempering temperatures on hardness of oil quenched SH737-2Cu-0.9C samples sintered at different temperatures

The hardness values in Table 5 and 6 show that the as-sintered samples have the same hardness as that achieved by brine quenching followed by tempering at 600 °C.

4. Conclusion

Sintered hardened grades have an advantage over simple carbon steels, as they do not require secondary processing, which is evident from the above data. The hardness of sintered SH737 samples is the same as that obtained from heat treatment and tempering, allowing the secondary hardness to take place. Sinter hardened steels have an enormous processing time advantage over simple carbon steels.

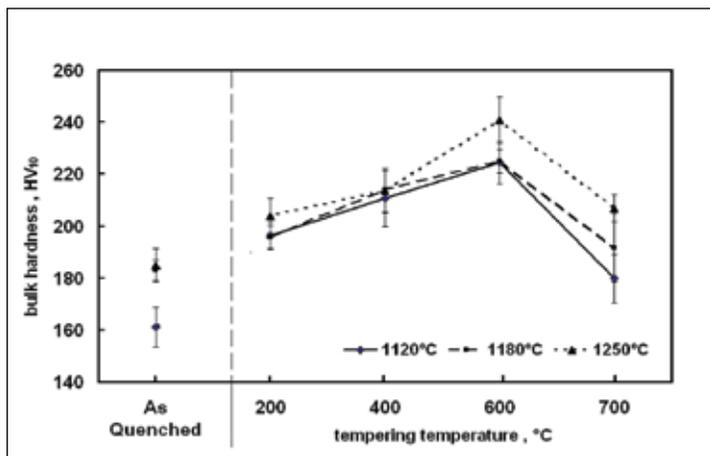


Figure 7. Effect of tempering temperatures on hardness of brine quenched SH737-2Cu-0.9C samples sintered at different temperatures

Table 5. Bulk hardness values of oil quenched samples at different tempering temperatures for SH737-2Cu-0.9C for different sintering temperatures

Bulk Hardness Values (HV ₁₀) for Oil Quenched + Tempered samples					
Sintering Temperature	As sintered hardness values	Tempering Temperature (°C)			
		200	400	600	700
1120°C	226 ± 4.3	166 ± 4.1	175 ± 4.7	200 ± 4.2	169 ± 3.8
1180°C	232 ± 9.4	167 ± 6.9	192 ± 3.2	202 ± 8.2	173 ± 6.3
1250°C	239 ± 4.9	174 ± 6.6	194 ± 4.4	204 ± 4.7	184 ± 4.0

Table 6. Bulk hardness values of brine quenched samples at different tempering temperatures for SH737-2Cu-0.9C for different sintering temperatures

Bulk Hardness Values (HV ₁₀) for Brine Quenched + Tempered samples					
Sintering Temperature	As sintered hardness values	Tempering Temperature (°C)			
		200	400	600	700
1120°C	226 ± 4.3	196 ± 5.1	211 ± 10.6	224 ± 8.3	180 ± 9.3
1180°C	232 ± 9.4	196 ± 4.6	214 ± 8.3	225 ± 4.6	192 ± 13.1
1250°C	239 ± 4.9	204 ± 6.8	214 ± 8.6	241 ± 9.0	207 ± 5.3

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Mr. Anand is currently pursuing his degree from the Department of Materials and Metallurgical Engineering at the Indian Institute of Technology, Kanpur. He has always been fascinated by the practical application of intellectual and conceptual knowl-

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Neerav Verma is a senior undergraduate student of Materials and Metallurgical engineering in Indian Institute of Technology, Kanpur, India. The versatile field of Material science has always captivated him and the research areas in it have inspired him to explore them and ameliorate his knowledge to come up with decent research work.

He has been involved in a rigorous research work in the field of Powder Metallurgy which included “Investigation Of Densification And Microstructural Evolution Of Sinter-Hardening Alloy Steels.” He has also worked in the field of characterization of Construction Materials during his 2006 summer internship at LMC Lab, EPFL, Lausanne, Switzerland.