CHARACTERIZATION OF SINTERED VALVE SEAT INSERTS OBTAINED WITH AISI M2 HIGH-SPEED STEEL AFTER AIR QUENCHING*

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Abstract
The aim of this work was to heat treat and characterize sintered valve seat inserts (VSI). The powder metallurgy route was the only way found to substitute cobalt and lead, used in the VSI original alloy, due to their high cost and toxicological effect, respectively. The studied VSI was obtained with AISI M2 high-speed steel powder mixed with iron powder and other additives such as manganese sulphide, graphite, zinc stearate, carbides and copper, which was added by metallic infiltration. All the VSI were air quenched and double tempered, for one hour each, at seven different equidistantly temperatures, ranging from 100 °C up to 700 °C. The physical and mechanical properties were evaluated by means of the VSI apparent density, apparent hardness and crush radial strength. The chemical composition was determined through gas analysis, for the light elements such as carbon and sulfur, and energy dispersive X-ray fluorescence spectrometry for other elements. Microstructural characterization was performed with the support of scanning electron microscopy and energy dispersive spectroscopy. Regarding the VSI final application, the best results were achieved with the inserts air quenched and double tempered at 600 °C.
Keywords: Powder metallurgy; Heat treatment; Valve seat insert; AISI M2 high-speed steel.

CARACTERIZAÇÃO DE INSERTOS PARA ASSENTOS DE VÁLVULAS SINTERIZADOS OBTIDOS COM O AÇO RÁPIDO AISI M2 DEPOIS DE TEMPERADOS AO AR

Resumo
O objetivo deste trabalho foi o de tratar termicamente e caracterizar os insertos para assento de válvula (do inglês VSI) sinterizados. A rota da metalurgia do pó foi a única maneira encontrada para substituir-se o cobalto e o chumbo, utilizados na liga original do VSI, devido ao seu alto custo e efeito toxicológico, respectivamente. Os VSI do presente trabalho foram obtidos com o pó de aço rápido AISI M2 misturado com pó de ferro e outros aditivos como sulfeto de manganês, grafite, estearato de zinco, carbonetos e cobre, que foi adicionado por infiltração metálica. Todos os VSI foram temperados ao ar e duplamente revenidos, por uma hora cada, em sete temperaturas equidistantemente diferentes, variando de 100 °C a 700 °C. As propriedades físicas e mecânicas foram avaliadas através da densidade aparente dos VSI, dureza aparente e resistência à ruptura radial. A composição química foi determinada pela análise de gás, para os elementos leves como carbono e enxofre, e espectrometria por energia dispersiva de fluorescência de raios X para os outros. A caracterização microestrutural foi realizada com o suporte da microscopia

eletrônica de varredura e espectroscopia de energia dispersiva. Em relação à aplicação final dos VSI, os melhores resultados foram alcançados com os componentes temperados ao ar e duplamente revenidos a 600 °C.
Palavras-chave: Metalurgia do pó; Tratamento térmico; Inserto para assento de válvula; Aço rápido AISI M2.

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1 INTRODUCTION

The powder metallurgy (P/M) process is a near-net or net-shape manufacturing process that combines the features of shape-making technology for powder compaction with the development of final material and design properties (physical and mechanical) during subsequent densification or consolidation process, e.g. sintering. It is critical to recognize this interrelationship at the outset of the design process because a subtle change in the manufacturing process can cause a significant change in material properties [1].
The mechanical set partially responsible for sealing the combustion chamber is constitute of valve and valve seat insert, as shown in Figure 1. These components represent a huge challenge, from the metallurgical point of view, when the aim is to increase the engine's performance and reduce its production cost [2-4].

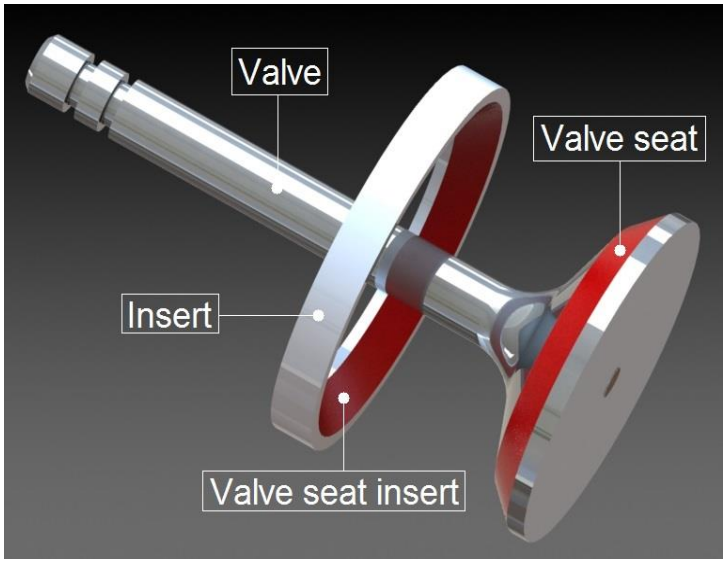

Figure 1. Drawing in perspective of the valve seat and valve seat insert positioning.

In terms of thermal cycle, the valve seat and valve seat insert operate under severe conditions, and there are two main critical situations corresponding to the air / fuel mixture intake and exhaust. During the intake of the gaseous mixture, the maximum temperature at the valve seat insert is around 250 °C, and the valve seat can hit up to 350 °C. The worst case occurs in the exhaust stroke when the temperature at the valve seat can heat up to 700 ºC, and at the valve seat insert up to 350 ºC [5].
Powder metallurgy processing offers several advantages to costly and highly alloyed tool steel materials. These advantages include uniform and finer microstructure, improved grindability, improved cutting performance, and capabilities of high-speed steels and tool steel alloys that cannot be made by conventional ingot metallurgy [1].
The aim of this work was to air quench, double temper in different temperatures, and characterize all the sintered valve seat inserts obtained with the powders mixtures of AISI M2 high-speed steel, iron, and others additives. Santos et al. [6-8] previously developed the studied VSI, and he has only evaluated the as sintered VSI.

2 MATERIALS AND METHODS

The VSI studied at the present work was obtained through the powder metallurgy route. The developed powders mixtures is mainly composed of AISI M2 high-speed

steel (HSS) and iron powders, there is also others additives such as manganese sulphide, niobium carbide, graphite and zinc stearate to improve the lubrication of the components during compaction. The chemical composition of such powders is shown in Table 1. Moreover, the AISI M2 HSS had its particle size distribution analyzed through the principle of laser multi angle analysis [9].

Table 1. Nominal composition (mass %) of the developed powders mixtures

Alloy / element	**AISI M2**	**Fe**	**MnS**	**NbC**	**C (graphite)**	**Zn stearate**	**Cu (infiltration)**
Alloy 1	43.6	43.6	0.5	2.0	0.3	0.8	10.0

All the powders mentioned in Table 1 were mixed in an intensive mixer for 300 s at 1,715 rpm, except copper because it was added by metallic infiltration, i.e., two compacts with dimensions of 32.5 x 25.5 x 5.9 mm^3 were pressed from the iron powders mixtures and cooper before the sintering process took place. These two compacts had to be put together, to mount one on top of the other. Green compacts were obtained from the powders mixtures compaction in a double action automated hydraulic press at a pressure of 700 MPa.

The compacted VSI was pre-heated up to 600 °C (0.275 °C/s) for 2,100 s to ensure the zinc stearate elimination. Then, it was sintered at 1,150 °C (0.228 °C/s) for 2,400 s. By last, the VSI was cooled until room temperature with a rate of 0.335 °C/s. The sintering process have been performed in a continuous commercial belt furnace under a hydrogen-based (90% H_2 + 10% N_2) atmosphere.

The sintered VSI heat treatment consisted of austenitizing it at 1,150 °C (heated at a rate of 0.480 °C/s) for 1,200 s in a laboratory muffle. In order to avoid decarburization, the VSI were wrapped in a blick white sulphite drawing paper and put inside a cast iron box containing a mixture of 50% C (graphite) + 50% Al_2O_3 (aluminum oxide). This box was then put inside the muffle for heat treatment. A thermocouple type k was attached to the samples and a data acquisition system was used to measure the cooling rate of the VSI samples. The austenitized samples were at that time air quenched until room temperature. All air quenched VSI were double tempered till room temperature, for one hour each at seven equidistant different temperatures, ranging from 100 °C up to 700 °C.

The components physical and mechanical properties were determined through three main tests. The apparent hardness of all heat treated VSI was determined in accordance with the standard ASTM E 10-01 [10]. This test method covers the determination of the Brinell hardness of metallic materials. Then, the as sintered densities were measured immersing the inserts into water using the Archimedes method, in agreement with the standard ASTM C 373-88 [11]. By last, the crush radial strength test was performed in agreement with the standard MPIF 35 [12]. This standard test consists of radially compress the VSI until its first cracks appear.

The chemical composition analyses were carried out using two techniques, gas analysis and energy dispersive X-ray fluorescence (EDXRF) spectrometry. The gas analyzer equipment determined the content of light elements, such as carbon and sulfur within the samples. This apparatus uses an induction furnace and measures the amount of each element by infrared absorption [13]. The others elements were measured using the EDXRF technique [14-16].

Microstructural characterization was first performed preparing the samples according to standard metallography procedures, i.e., mounting, grinding, polishing and etching. All the samples were analyzed with the support of scanning electron microscopy

(SEM) and energy dispersive spectroscopy (EDS) for elemental identification. Before the specimens were analyzed, they were etched with Nital 3% (97% ethyl alcohol + 3% concentrated HNO_3) for 50 s [17].

3 RESULTS AND DISCUSSION

In mold filling, particle size distribution and particle shape are controlling factors in determining tap density of the filled mold [1]. The AISI M2 high-speed steel (HSS) particle size distribution is shown in Table 2.

Table 2. Particle size distribution of the AISI M2 high-speed steel powder

Alloy / parameter (µm)	**Diameter at 10%**	**Diameter at 50%**	**Diameter at 90%**	**Mean diameter**
AISI M2	21.82	60.33	194.84	90.10

The AISI M2 HSS powder was produced by gas atomization [18], therefore the HSS powder has a spherical shape as can be seen in Figure 2.

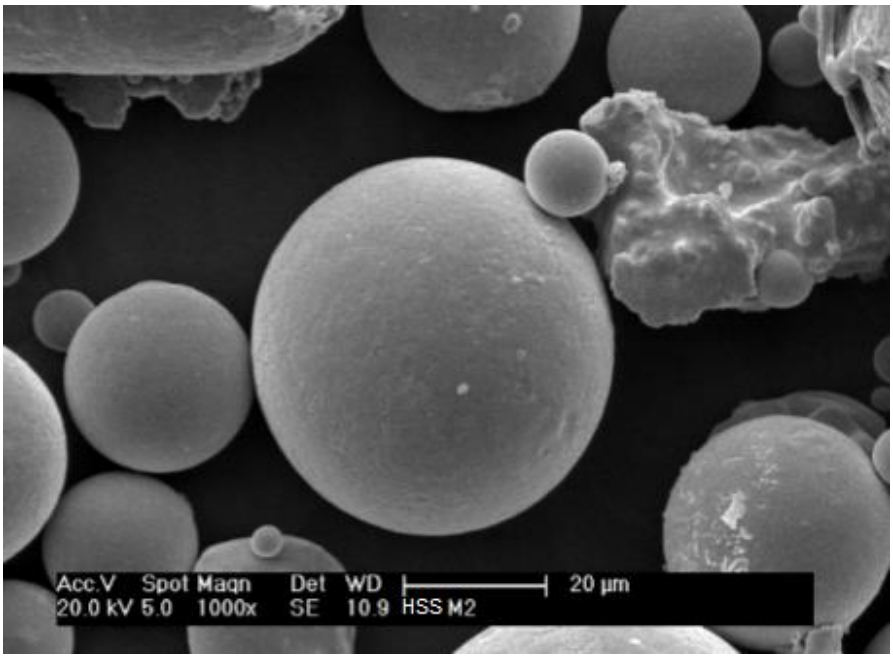

Figure 2. SEM micrograph of the AISI M2 HSS powder.

The VSI apparent hardness was determined according to the Brinell model with a sphere of Ø 2.5 mm and a load of 187.5 kgf. Moreover, it can be represented as an international standard abbreviation by HB 2.5 / 187.5. The hardness measurement was done for the VSI as sintered and heat treated ones, which were air quenched and double tempered, one hour each, at different temperatures ranging from 100 °C up to 700 °C. This hardness variation is shown in Table 3.

Table 3. Brinell hardness (HB 2.5/187.5) variation for the VSI as sintered, quenched in air (from 1,150 ˚C) and double tempered at seven equidistant different temperatures

	Tempering temperature (°C)							
	As sintered	**100**	**200**	**300**	**400**	**500**	**600**	**700**
Alloy 1	366 ± 4	345 ± 5	380 ± 5	327 ± 3	318 ± 2	425 ± 8	392 ± 1	299 ± 1

The hardness requirements, for the VSI final commercial application, according to the automakers should be between 370 - 410 (HB 2.5 / 187.5). Table 3 also shows that the heat treatment that most got close to the established hardness values is Alloy 1 (AISI M2 powder mixture) double tempered at 600 °C. Therefore, it worth mention that all the following given results and discussion of the present work are focused on this heat treatment.

Although the apparent hardness is a very important property, the apparent density and crush radial strength (CRS) are as important as the VSI apparent hardness. Briefly, the results of the properties mentioned previously is shown in Table 4.

Table. Physical and mechanical properties of the valve seat insert obtained with Alloy 1 (AISI M2 powder mixture) air quenched and double tempered at 600 °C

Alloy / property	**Apparent density** (g/cm^3)	**Hardness** (HB 2.5/187.5)	**Crush radial strength** (MPa)
Alloy 1	7.4 ± 0.3	392 ± 1	579 ± 92

In comparison with the work developed previously by Santos et al. [6-8], only with as sintered components, the apparent density can be considered the same, 7.4 g/cm^3. The apparent hardness values measured by Santos [7] was lower, 345 ± 21 HB, than the one obtained in the present work, 392 ± 1 HB. Additionally, the CRS values obtained by Santos [7] was a little bit higher, 595 ± 48 MPa, than that shown here, 579 ± 92 MPa. Although the CRS values obtained in the present work are lower when comparing to that one developed by Santos [7], this property is only necessary when assembling the VSI into the engine block. Therefore, there is no minimum level requirement for the CRS, the component just need to withstand the assembly step.

The measured chemical composition of the VSI in shown in Table 5. The carbon and sulfur contents were determined through the gas analysis method; all the other elements were determined using energy dispersive X-ray fluorescence (EDXRF) spectrometry.

Table 5. Valve seat insert chemical composition (mass %) determined by gas analysis and energy dispersive X-ray fluorescence (EDXRF) spectrometry. The carbon and sulfur contents were determined using gas analysis.

Element	**Fe**	**Cu**	**Mo**	**W**	**Cr**	**Nb**	**V**	**C**	**Mn**	**Si**	**S**
Alloy 1	73.52 ± 0.02	15.04 ± 0.01	2.77 ± 0.01	1.62 ± 0.01	2.09 ± 0.01	2.03 ± 0.03	0.79 ± 0.01	0.97 ± 0.03	0.78 ± 0.06	0.27 ± 0.07	0.12 ± 0.02

Regarding the copper concentration, shown in Table 5, it can be noticed a considerable variation from its nominal value (see Table 1). The copper value should be 10%, but the measured value was 15.04%. Such copper variation can be correlated by not precisely measuring the copper ring's mass, which should be ten percent of the insert's mass, i.e., the insert and the copper ring should be precisely paired.

The cooling rate during air quenching for the studied VSI was measured with a thermocouple type k attached to the sample and to a data acquisition system, and it was 0.575 °C/s. Figure 3 shows the elemental distribution for the AISI M2 HSS obtained by SEM with elements identification using EDS. It was necessary to over etch the samples, only doing it was possible to differentiate between the HSS matrix and the iron one, because both were added at similar amount (see Table 1) and reacts in a different way by the same etchant. After over etching the sample, phases

such as bainite (B, light gray), ferrite (Fe, gray) and martensite having a high carbon content (M', dark gray) seemed to be corroded. Others elements such as niobium carbide (NbC, white) and manganese sulfide (MnS, very dark gray) can be easily distinguished over the microstructure. In addition, it can be seen in Figure 3 a lower amount of copper when comparing to Table 5, and it may attributed to a particular region of the sample.

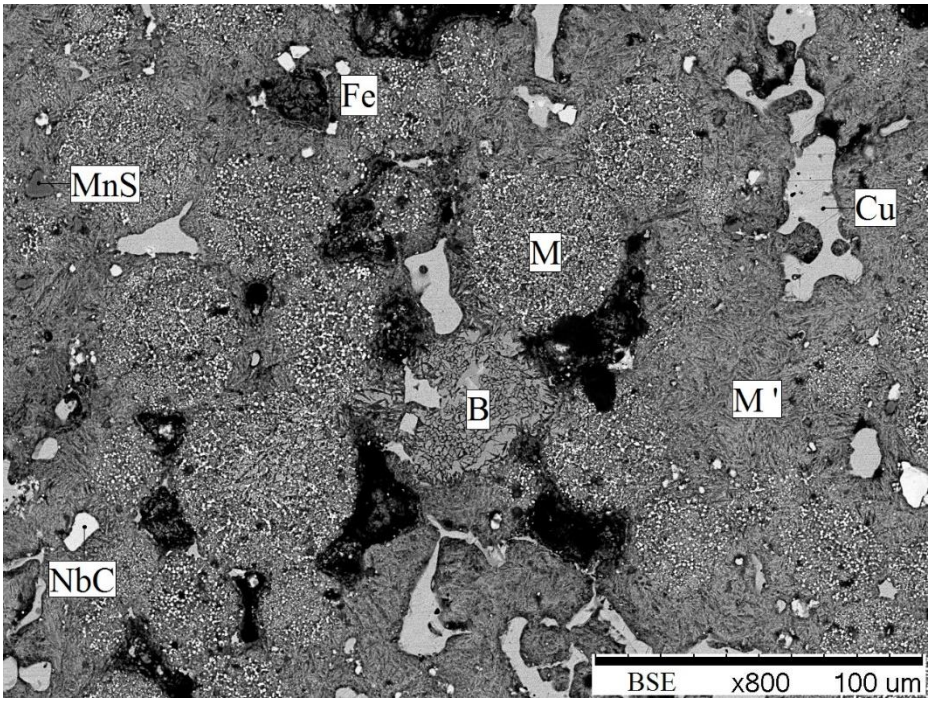

Figure 4. SEM micrograph with elemental identification using EDS of Alloy 1 (AISI M2 powder mixture) air quenched and double tempered at 600 °C.

4 CONCLUSION

The air quenching, despite the low cooling rate, gave the desired variation of properties and phase transformations.
The values of hardness tends to decrease when the measured crush radial strength increases.
Despite the many different phases present at the microstructure, the AISI M2 high-speed steel seems to be the element with most influence on it, thus, keeping the good properties of the components.

ACKNOWLODGMENTS

The authors want to thank CAPES and CNPq for the financial support. In addition, General Motors South America, Höganäs and Group Combustol & Metalpó for materials and equipment supply.

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