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Effect of Homogenization &Quenching Media on the Mechanical Properties of Sintered Hot Forged AISI 9250 P/MSteel Preforms

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ABSTRACT: *Present investigation is an attempt to generate experimental data in order to establish the influence of homogenization and cooling media on the mechanical properties of hot forged AISI 9250 sintered P/M steel to square cross-section bars produced using elemental powders. The elemental powders corresponding to final AISI 9250 composition ofFe-0.5%C-0.75%Mn-2.0%Si were taken in an appropriate proportion and blended in a pot mill for a period of 32 hours while maintaining the powder to ball ratio by weight as 1.1:1. Compacts of 28mm diameter and 36mm height were prepared on a 1.0 MN capacity Universal Testing Machine (UTM) and using suitable die, punch and bottom insert assembly in the density range of 85±1 percent of theoretical by applying the pressure in the range of 550±10 MPa and by taking pre-weighed powder blend. In all 36 compacts were prepared. These green compacts were coated using the indigenously developed ceramic coating and the same was allowed to dry under the ambient conditions for a period of 14-16 hours. These ceramic coated compacts were re-coated 90° to the previous coating and re-dried under the aforementioned conditions for the same length of time. Ceramic coated compacts were sintered in an electric muffle furnace at 1120±10°C for a period of 120 minutes and subsequently hot forged to square cross-section bars of approximate dimensions of 14mm X 14mm X 95-100mm on a friction screw press of 1.00MN capacity at the sintering temperature itself. Nine forged specimens were oil quenched and remaining 27 forged bars were homogenized at the sintering temperature for a period of 60 minutes followed by quenching nine of them in oil, nine specimens in air and remaining nine were cooled in the furnace itself. Standard tensile specimens were prepared from each set and tested for evaluation of mechanical properties followed by SEM Fractography on the fractured specimen surfaces. Tensile tests results have established that the homogenization step followed by cooling them in different media has improved the values of toughness as the per cent elongation and per cent area reduction, both, have gone up though the strength values have gone down.*

Key Words: Bars, Ceramic, Coated, Compacts, Density, Forged, Fractographs, Generate, Properties, Tensile.

I. INTRODUCTION

It is interesting to note that the period falling between late 1970s and the early 1980s have gone through a significant metallurgical break - through in the recognition of powder metallurgy (P/M)techniques principally in the form of the production of P/Mtools and P/Mforgings [1-14]. However, in the present scenario, the commercial powder metallurgy spans the density spectrum from highly porous metal filters through self-lubricating bearings and the P/Mparts with controlled density to fully dense P/Mwrought metal stems. But, today's P/M has crossed many hurdles and has become a very rapid, economical and high volume production methods for making precision components from elemental or alloy powders or elemental powder blends with or without the addition of carbide, ceramic, oxides, borides or nitrides etc. The technology of powder forging has established itself for fabricating powders into precise engineering parts which have properties comparable or even superior to those of conventional forgings [15-30]. The strength and the toughness of a forged metal assures a greater safety, whenever, the forged products are subjected to excessive loads and the internal stresses from the loads during actual service conditions [4, 5, and 15]. Apart from all these, the powder forging is an attractive manufacturing route for many components because of the fact that the material utilization is better than what is in conventional forgings. Further, the details and tolerances obtainable lead to the elimination of much, if not all finish machining and also induce tight tolerance in the as forged condition. The process technically sounds economical on the ground that lower forging temperatures are anticipated which would result in better surface finish and that would enhance the fatigue strengths [6, 28]. In addition to this, powder forging requires one or two blows instead of three or many more that are necessarily required in the conventional forging of bar stocks. Single quality of iron powder can be used to produce wide variety of steels of any desired composition. Reduced die wear and simplified die configuration accompanied by the expected properties that can be successfully tailored to the required applications. Basically, these factors govern the economics of the process, favouring ultimately the use of powder preform forgings [31-42], therefore, every forging operation must have definite requirements or engineering specifications with the intended applications. The mechanical properties of these forgings are normally determined from the test pieces that are taken from the forgings themselves or other from locations well within the body of the forgings or full size prolongations provided for this purpose [7,8,11-15,18,19,27]. However, technically, the forging of sintered P/Mpreforms whether it is cold, warm or hot, involves three main stages of deformations, namely, upsetting, plane - strain and hydrostatic as reported elsewhere [43-46]. In the above stages, the densification as well as shape changes do occur simultaneously, and, therefore, it is, fundamentally essential to assess the mechanical properties such as the tensile strength, the fracture strength, the hardness, the per cent elongation and the per cent area reduction of the final product. The present investigation is also aimed to assess the mode of fracture during tensile testing of specimens subjected to different heat treatments along with the tensile properties evaluations. Thus, it is obvious that the mechanical properties of the product must depend upon the modefollowed to achieve the complete densification as well as the shape change. Obviously, the densification must precede the shape change or otherwise the component would remain porous. This effect is anticipated to be detrimental to the expected mechanical properties of the P/M forged parts. However, for clarity to be demonstrated, fig. 1 shows the influence of porosity on mechanical properties of the P/M parts. Fig. 1 very clearly shows that as the porosity content in P/M parts are increased, all tensile and impact properties are abruptly dropped. Therefore, in the present investigation attempt is made to ensure that the production of fully dense products and their properties are assessed in clear terms. Thus, from this fig. 1, it is very clear that if the P/Mproduct is to be developed for high load carrying capacity in the stressed conditions, it is, therefore, imperative to reduce the porosity content to almost nil level. It has been reported that the porosity levels can be brought down to almost zero level by means of applying adequate pressures at elevated temperatures [29, 30]. It is also reported [47] that at sometimes an additional impact load is required so as to create sound metallurgical bond across the collapsing pore surfaces. Tensile properties of P/M parts can be referred elsewhere [7,11,13-15,17-19,31,35,38,39,41,42,47]. Literature reporting mechanical properties of P/Mforged parts such as fatigue and impact can be referred elsewhere [4- 6, 26, 28]. However, the properties such as ring rupture strengths can be found elsewhere [48-50]. Now, therefore, in the present investigation, an attempt is made to develop an AISI 9250 grade of sintered hot forged P/Msteel using elemental powders.

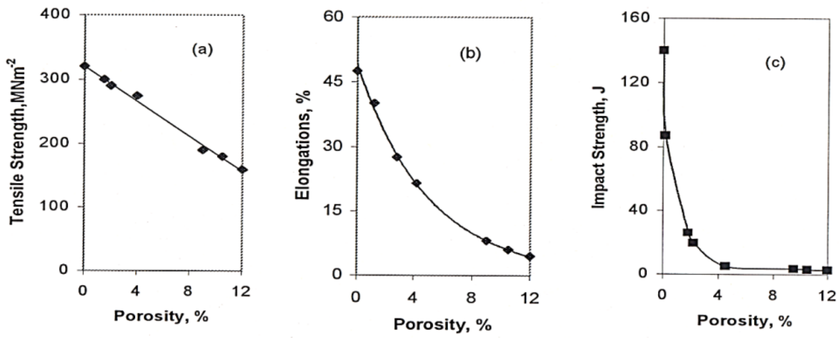

Figure 1 Mechanical Properties of Powder Metallurgy Components against Porosity

II. EXPERIMENTAL DETAILS

Atomized iron powder of -180 ×μm with the chemical purity of 99.63 per cent was procured from the M/s.Sundaram Fasteners, Hyderabad, A.P., and India. This powder contained 0.37 percent insoluble impurities. Manganese and Silicon powders were obtained from the M/s. The Ghrishma Specialty Powders, Mumbai, Maharashtra, India. All these powders were of the particle size below 37μ. However, the graphite powder of 2-5 μm was supplied by the M/s. Asbury Graphite Mills, Inc., Warren County, New Jersey, U.S.A. The basic characteristic features of the iron powder and powder blend prepared to yield the composition of AISI 9250 P/M steel are given in Table I and Table II respectively.

Table I Basic Characteristic Features of Iron Powder and AISI 9250 Steel Blend

Sl. No.	Property	Iron Powder	AISI 9250 Blend
1.	Apparent Density, g/cc	2.93	3.24
2.	Flow rate by Hall Flow Meter, Sec./50g	24.10	23.64
3.	Compressibility, g/cc at a pressure of 410±10 MPa	6.4	6.37

Table II Sieve Size Analysis of Iron Powder

Sieve Size, μm	% Weight Retained	Cum. Wt.% Retained
-180 + 150	3.61	3.61
-150 + 125	3.62	7.23
-125 + 106	2.48	9.71
-106 + 90	0.70	10.41
-90 + 75	8.33	9.20
-75 + 63	9.20	27.94
-63 + 53	16.68	44.62
-53 + 45	15.83	60.45
-45 + 37	3.60	64.05
- 37	35.89	99.94

II.1 Powder Blend Preparation

Required amounts of iron powders of size -180 μm, manganese (-37μm), silicon (-37μm) and graphite (2-5μm) were accurately weighed and mixed in a stainless steel pot with a powder mix weight to porcelain balls (12-19mm diameters) weight in the ratio of 1.1: 1 and the pot lid was securely tightened. The blending operation was carried out on a pot mill for a period of 32 hours. Uniformity in blending was tested as per the procedure described elsewhere [36-39]. Once the blending operation was completed, the porcelain balls were removed manually from the powder blend and the blend was available for preparing the green compacts of suitable initial aspect ratios with desired initial preform geometries and the densities.

II.2 Compact Preparation

Green compacts (28 mm diameter and 35 mm height) of powder blend corresponding to AISI 9250 steel were prepared on a 1.0 MN capacity Universal Testing Machineusing a suitable die, punch and the bottom insert along with top and bottom die plates. During compaction graphite powder paste with acetone was used as a lubricant to minimize friction between the inner die walls, punch and bottom insert surfaces coming in contactwith the powder blend charge in order to minimize the friction between the powder blend charge and the die, punch and the bottom insert surfaces. Further, in order to maintain the constant aspect ratio (height to diameter ratio), a pressure in the range of 540±10 MPa was employed while taking the accurately pre-weighed powder blend. Thus, the initial compact density was maintained in the range of 85±1 per cent of theoretical. Fig. 2 shows the die, punch, thebottom insert and the powder blend compaction assembly.

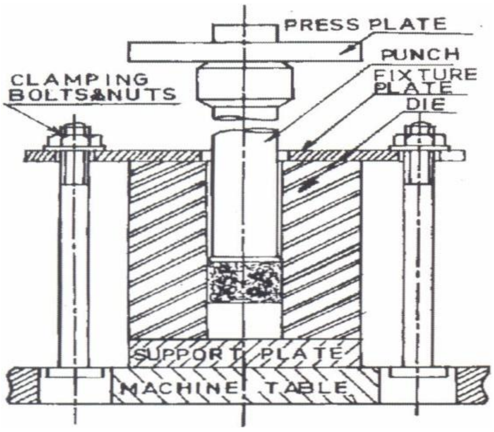

Figure 2Die, Punch and the Bottom Insert Compaction Assembly along with the Powder Blend.

II.3 Application of Indigenously Developed Ceramic Coating

Indigenously developed ceramic coating [51] was applied over the entire surfaces of the green compacts and then the coating was allowed to dry under the ambient conditions for a period of 16 hours. Immediately after the drying of this applied ceramic coating, all these compacts were recoated 90° to the previous coating and the same were allowed todry once again under the aforementioned conditions. The ceramic coating, thus, applied protects the compacts from oxidation and subsequent loss of carbon. The ceramic coating employed in the present investigation was tested up to 1250 ± 10 °C. It was found that this coating had successfully protected the compacts during the sintering operation.

II.4 Sintering and Hot Forging to Square Cross-Section Bars

Indigenously developed ceramic coated compacts were sintered in an electric muffle furnace at 1150±10^{0}C for a period of 120 minutes. Once the sintering schedule was completed, the sintered compacts were hot forged to square cross-section bars of approximate dimensions of 14mm x 14mm x 95-l00 mm. During forging operation care was taken to avoid excessive drop in the forging temperature by reheating the partially upset forged preforms to the sintering temperature and retaining them at this temperature at least for 30 minutes before re-forging them. This provided the quality forged products. In addition to the above, the upset die set assembly was pre-heated in the temperature range of 350 ±10°C to avoid excessive die chilling. The hot forging operation was carried out on a friction screw press of 1.0 MN capacity. Once the forging operation was completed, nine forged square cross-section bar specimens were oil quenched and the remaining twenty seven forged bars were homogenized in the furnace itself for a period of 1 hour at 1150±10^{0}C. Immediately after the completion of homogenizing schedule, nine bars were oil quenched, six specimens were air cooled and the remaining nine bars were cooled in the furnace itself. Thus, four groups of specimens were categorized such as: (A) Sintered - Forged and Oil Quenched (SFOQ); (B) Sintered - Forged - Homogenized and Oil Quenched (SFHOQ); (C) Sintered - Forged - Homogenized andAir Cooled (SFHAC) and (D) Sintered-Forged-Homogenized and Furnace Cooled (SFHFC) for tensile testing. Tensile Testing. Five of thenine bars of each groups A, B, C and D were machined to a standard tensile test specimens for carrying out tensile testing.The tensile tests were carried out following the procedure described elsewhere [52]. Properties, such as tensile strength, the fracture strength, the percent elongation and the percent area reduction were calculated for all the specimens and the average values were taken in each group as the final. However, before carrying out the tensile tests, the density measurements were carried out for all the specimens. Density for each specimen was found to be virtually 100%.

III. RESULTS AND DISCUSSION

III.1 Tensile Properties Assessment

Table III shows the mechanical properties of sintered - hot forged and heat treated under different conditions for AISI 9250 P/Msteel prepared from the elemental powders and hot forged to square cross-section bars. The properties reported in this table include tensile strength, the fracture strength, the percentage elongation, the percentage area reduction, and the hardness values. It is, observed from this table that the sintered forged and oil quenched specimens have shown the maximum tensile strength of 1455 MPa. But, if this table is examined w.r.t. the processing such as (A) SFOQ, (B) SFHOQ, (C) SFHAC and (D) SFHFC, it is noticed that the ultimate tensile strength has dropped and the true fracture strength values have gone up. Except in the case SFHOQ condition where there is a little drop in the value In general, the percent elongation and the per cent area reduction have shown an increasing trend in order of consideration, i.e., (A), (B), (C) and (D) respectively. The hardness values have also dropped in the same sequence.

Table III Mechanical Properties of AISI 9250 Sintered - Hot Forged and Heat Treated P/M Steel

Condition	UTS, MPa	True Fracture Stress, MPa	Engineering Fracture Stress, MPa	% El.	%A.R.	Hardness Vickers, HV.
SFOQ	**1455**	**1480**	**1457.8**	**4.80**	**l.50**	**445**
SFHOQ	**968**	**1050**	**952.88**	**16.60**	**9.25**	**311**
SFHAC	**952**	**1093**	**982.39**	**17.04**	**10.12**	**309**
SFHFC	**728**	**1137**	**907.21**	**20.21**	**14.34**	**290**

III.2 Relative Tensile Test Results of AISI 9250 Forged P/M Steel

Fig. 3 shows the stress versus per cent elongation curves (only a relative magnitude, not the actual units) for the sintered hot forged AISI 9250 P/Msteels heat treated under the conditions described by the abbreviations SFOQ,SFHOQ, SFHAC and SFHFC respectively. None of these curves have exhibited distinct yield points (either lower orupper).It is further observed that the AISI 9250 steel under SFOQ condition has

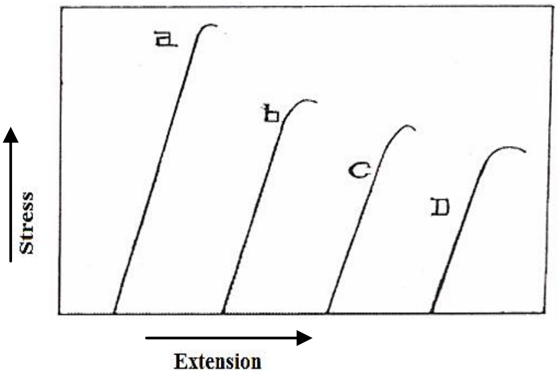

Figure 3Relative Plots Between Stress and Percentage Elongation for the specimens

(a) SFOQ = SinteredForged & Oil Quenched(A);(b) SFHOQ = Sintered Forged Homogenized for 1 hour at 1150± 10°C and Oil Quenched (B); (c) SFHAC = SinteredForged Homogenized for 1 hour at 1150± 10°C and Air Cooled (C) and (d) SFHFC = Sintered Forged Homogenized for I hour at 1150± 10°C and Furnace Cooled (D))

exhibited highest UTS and highest true fracture strength values, but, with the least values of percent elongation and the percent area reduction. The relative appearance of the stress versus percentage elongation curves for each heat treatment condition corresponded very well to the calculated values that are shown in Table III.

III.3 Evaluations of SEM Fractographs

III.3.1 SEM Fractograph of Tension Tested AISI 9250 Steel under SFOQ and SFHOQ Conditions

Fig.4 shows the scanning electron fractograph of tension tested specimen corresponding to sintered and hot forged but oil quenched condition. The fractograph shows the failure occurring in a typical way resembling to a quasi- cleavage fracture. The structure also shows few micro-cracks and presence of porosity. Therefore, the fracture mode is associated to be highly brittle with a negligible degree of ductile failure. Few dimples do indicate the fracture to be partially associated to be ductile. Hence, it is established that the failure was truly brittle and insignificantly ductile. Fig. 5 demonstrates the SEM fractograph of tension tested specimen of AISI 9250 sintered - hot forged homogenized at 1150±10^0 C for a period of 1 hour and oil quenched. At 1000X magnification, fine dimples are seen throughout the fractographand at couple of places well grown voids do indicate that there had been a clear case of coalescence of pores into a fully blown voids - resulting into failure of the specimen. Thus, the fracture mode is characterized by mainly ductile and partly brittle which can be further called as mixed mode.

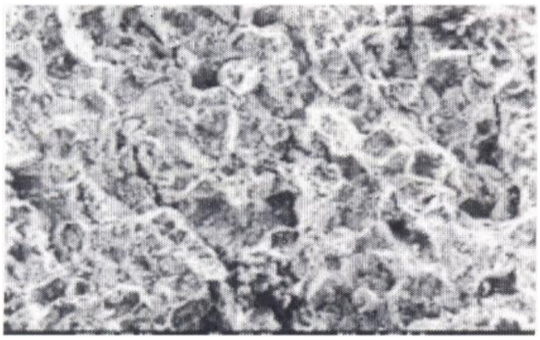

Figure 4 Fractograph of AISI 9250 P/MSteel under SFOQ

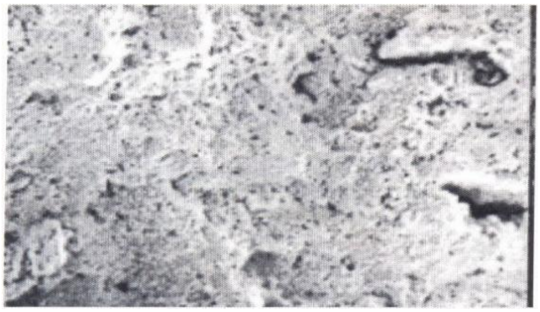

Figure 5 Fractograph of AISI 9250 P/MSteel under SFHOQ

III.3.2 SEM Fractograph of Tension Tested AISI 9250 Steel under SFHACand SFHFC Conditions

Fig. 6 shows the SEM fractograph of tensile fractured specimen of hot forged - homogenized at 1150±10° C for a period of 1 hour and cooled in still air. This fractograph exhibits fairly distributed fine dimples and at places a coalescence of pores while the fracture proceeded. At places, a sign of tearing is also evident. The fractograph has shown an overall picture of being fairly ductile and partially brittle. This is in close conformity with the experimentalresults of tensile testing showing over 17 per cent elongation and over 10 per cent area reduction. Thus, the failure mode is characterized to be extensively ductile and partially brittle. This is a mixed mode of failure. Incidental presence of porosity in the structure is virtually absent.

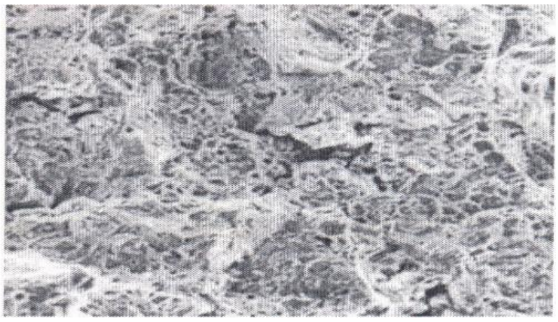

Figure 6Fractograph of AISI 9250 P/Steel under SFHAC

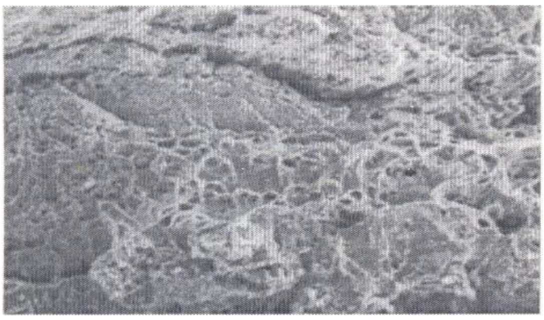

Figure 7 Fractograph of AISI 9250P/MSteel under SFHFC

Fig. 7 shows the fractograph obtained through the scanning electron microscope from the fractured surface of tensile tested specimen which was machined from the sintered - hot forged square cross-section and the same was homogenized for 1 hour at 1150±10°C and subsequently cooled inside the furnace itself. The fractograph is taken at 1000X magnification. This fractograph predominantly exhibits the large number of dimples and also at places coalescence of pores while the structure started showing failure. The mode of failure is highly ductile which is in tune with the percent elongation of 20.21 and percentage area reduction of 14.34 respectively.

IV. CONCLUSION

Based on the critical analysis of the experimental data and the calculated parameters, along with the analysis of the Fractographs of the tensile tested specimens under all the conditions of heat treatments, the following salient features as major findings emerged out from the present investigation and they are as beneath:

1. Mechanical properties such as UTS and True F.S. have been found to be maximum under SFOQ condition and minimum under SFHFC condition of heat treatment. However, the percentage elongation and the percentage area reduction followed the increasing trend in a sequence such as SFOQ, SFHOQ, SFHAC and SFHFC respectively. It is also established that the UTS and true FS values were found to decrease in the same order of sequence, i.e., SFOQ, SFHOQ, SFHAC and SFHFC,
2. The failure mode established during tension testing under SFOQ condition to be highly brittle and virtually no trace is noticed to establish that this failure mode to be ductile. Hence, the failure mode ispredominantly characterized as brittle mode of failure,
3. The failure mode during tensile testing under SFHOQ is characterized to be mainly ductile and partly brittle,
4. Failure modes under SFHAC and SFHFC conditions have been established to be predominantly ductile. However, traces are also present in the structure to associate the failure to be mildly brittle and predominantly ductile.

Summing up, it is concluded that the heat treatment schedules such as homogenizing the forged bars at 1150± 10°C for a period of 1 hour followed by quenching them in oil or in still air or cooling in the furnace itself has resulted in a substantial drop in ultimate tensile and fracture strength values coupled with the enhancement in per cent elongation and per cent area reduction, i.e., an increasing trend in toughness. Thus, the present investigation has established the feasibility of producing successfully an AISI 9250 P/Msteel by using elemental powders for structural applications. Many more such AISI grade of P/M steel can be successfully produced by using elemental powders which may possess strong potential for structural applications.

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