Development of high strength iron–phosphorus based P/M alloys

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Mechanical tests were conducted on iron–phosphorus powder alloys which were made using a hot powder forging technique. The technique involved hot forging of mild steel encapsulated powders. After forging the powders into slabs, the slabs were hot rolled and annealed to relieve the residual stresses. The alloys were studied in terms of microstructure, porosity content/densification, hardness and tensile properties. The porosity content was as low as 1.44% in one of the alloys. Microstructures of these alloys consist of single-phase ferrite only. The alloys, i.e. Fe–0.35P–2Cu–2Ni–1Si–0.5Mo and Fe–0.35P–2Cu–2Ni–1Si–0.5Mo–0.15C showed high strength. In the present study, it was observed that the alloying additions, such as Si, Mo, Ni and C to Fe–P based alloys caused an increase in strength along with reduction in ductility. Cu was added to reduce porosity of Fe–P alloys. Alloys developed in the present study were capable of being hot worked to very thin gauge of sheets and wires.

Keywords: Fe–P alloys, metallurgical process, P/M alloys, phosphorus.

PHOSPHORUS in steel in general increases the yield strength, ultimate tensile strength and hardness, but decreases both elongation and reduction in area at failure. Phosphorus contents however promote brittle behaviour in wrought route owing to segregation of Fe_3P along grainboundries[1].

Phosphorus also promotes cold short behaviour. However phosphoric iron can be worked plastically at ambient temperatures provided it is not exposed to very low temperatures, high loading rates, does not possess specimen geometries which cause local stress concentration and high carbon contents[1].

Phosphoric irons can be easily hot forged and also cold worked under suitable conditions, like low strain rates and with geometries that avoid stress concentration. Stewart *et al.* noticed that Fe–P alloys could be easily hot forged at 900°C compared to Fe–C alloys, which was explained partly to be due to stabilization of ferrite to high temperatures and the known fact that the hardness of ferrite drops rapidly below that of austenite at high temperatures. Moreover, the pearlitic transformation in Fe–C alloys limited the temperature below which forging was feasible[2].

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In recent years there has been an increasing interest in phosphorus-containing sintered alloys (PSAs). Phosphorus is added to P/M materials to improve a number of their characteristics or to reduce the cost of manufacturing without adversely affecting their properties. Powder processing of Fe–P based alloys ensures higher level of phosphorus in solution with iron, thereby making it possible to hot and cold work unlike their wrought counterparts.

In wrought metallurgy, phosphorus is treated as an impurity because in cast steels, phosphorus exhibits strong segregation during solidification, with the formation of inclusions at grain boundaries which lead to embrittlement. Because of this, in the majority of steels its amount does not exceed 0.05%. Cases where certain properties of steels and cast irons are improved by higher phosphorus contents are extremely rare[3].

The main advantages of phosphorus as an alloying element in powder metallurgy are: its ability to form with metals eutectics of relatively low melting points (970, 1150, 1300 and 1048 for the Cu–P, Ni–P, Co–P and Fe–P systems respectively) characterized by good fluidity and adhesion to metals and many refractory compounds; high diffusional mobility of its atoms in metals; and its ability to precipitation-harden metals and comparatively low cost[4].

Near full density pure Fe P/M parts can be very easily manufactured using conventional powder metallurgical process. It was observed that partial replacement of silicon by a small amount of phosphorus will activate sintering process in Fe–Si alloys by the formation of low melting eutectic phase with iron. Copper in Fe–P alloy helps to reduce porosity. Molybdenum, nickel alloying element improves strength of Fe–P alloy. Small amount of carbon is helpful to drive out phosphorus from grain boundaries. Phosphorous also helps carrying alloy constituents into iron matrix which are otherwise sluggish to diffuse. Phosphorus significantly improves ductility and strength of Fe–P based powder alloys[5]. It is therefore, realized that, the Fe–P based alloys, containing alloying elements, such as silicon, molybdenum and nickel could be used for structural application because of their higher strength than pure iron with reasonably good ductility. Since all these alloying elements (except nickel) are ferrite stabilizers, ferrite phase will be stable even at high temperature when substantial alloying is completed. Self-diffusion coefficient of iron[6] and inter-diffusion coefficient of the alloying elements in ferrite is much higher than that in austenite. This diffusion helps in reducing amount of pores in the P/M part. However, during alloying process some additional pores may be created[6] (due to dissolution of elemental particles).

High temperature processing (>1100°C) promotes diffusion. A reducing atmosphere removes oxide layer and improves particle bonding and improves cleanliness of the P/M part. It is therefore realized that application of

high temperature and use of reducing atmosphere favours the densification process and cleanliness of Fe–P based alloys and improves the mechanical properties of these alloys. However, if we follow the traditional powder metallurgical process, such as compaction and sintering, for manufacturing high phosphorous Fe–P based alloys, heavy volume shrinkage will be experienced[7]. Therefore, phosphorous higher than 0.6 wt% is not normally recommended in conventional P/M process involving compacting and sintering. There are several other densification processes available in the literature. Out of all densification processes available, hot iso-static pressing (HIP) is the best as far as density and performance of these P/M parts are concerned. However, the process is extremely costly. Therefore, some pseudo-HIP processing could be used for manufacturing these alloys to reduce the cost of processing without sacrificing the benefit of HIP processing. However, HIP process does not have scope of cleaning particle surfaces during processing. Furthermore, existence of prior particle boundaries (PPB) renders them unsuitable because PPBs are the source of impurity concentration resulting in inter-particle brittle failure. In view of this, in the present investigation, the densification of the Fe–P based alloys were carried out by hot powder forging[8] technique. Volume shrinkage associated with these alloys is also no more a consideration in hot powder forging. Hot powder forging has another feature which is not available with compacting in a die or HIP. It is essentially the process where shaping and consolidation are deformation based. This causes redistribution of residual impurities if any, situated at the particle surfaces and renders improvement in properties of the final product[9].

For making Fe–P, Fe–P–Cu–Mo–Ni–Si (with or without carbon) alloys by powder metallurgical technique, ferro-phosphorus, ferro–silicon, ferro–molybdenum powders were prepared separately by grinding lumps of ferro–phosphorus, ferro–silicon, ferro–molybdenum (contains 22% P, 70% Si, 60% Mo respectively) with the help of mortar and pestle (iron) or filing. Powders with –75 μm size were employed for preparation of alloys. Pure copper and nickel powders having –75 μm size were taken for preparation of these alloys.

The powder blends were manually mixed to obtain the desired alloy chemistry. About 500 g of each blended mixture was then poured into a mild steel capsule (as shown in Figure 1). The encapsulated powders were heated in a tubular furnace at 1150°C for 45 min in dry hydrogen atmosphere in order to remove the oxide layer from the surfaces of the powder particles. Heated capsules were then forged with a 100 T capacity friction screw press to make slabs using a channel die. Three P/M alloys were made in the present investigation. These are (a) Fe–0.35 wt% P alloy; (b) Fe–0.35 wt% P–2 wt% Cu–2 wt% Ni–1 wt% Si–0.5 wt% Mo alloy and (c) Fe–0.35 wt% P–2 wt% Cu–2 wt% Ni–1 wt% Si–0.5 wt% Mo–0.15 wt% C alloy.

The compositions of these alloys are based on the powder mixture. Figure 2 illustrates the process of making slabs through hot powder forging technique. The slabs were then homogenized at 1200°C for 2–3 h depending on the alloy composition to eliminate compositional inhomogeneity. Silicon containing alloys were heated for 3 h whereas alloys without silicon content were heated for 2 h. This is because diffusion of silicon in iron is much slower than the other alloying elements. Mild steel encapsulation was then removed by machining. The slabs, after removal of mild steel skin, were hot rolled using flat roll and section roll at 900°C to make thin sheets and wires respectively. Rolling was carried out slowly at 900°C with 0.1 mm thickness/diameter reduction per pass. The rolling was done using small laboratory scale rolling mill with 10 cm roll diameter. The sheets and wires were then vacuum annealed at 950°C for 40 min to relieve the residual stresses. All the samples prepared this way were characterized in terms of density, microstructure, hardness and tensile properties as detailed here.

Homogenized slabs as well as hot rolled and annealed sheets and wires were subjected to metallographic examinations for determining porosity shown in Table 1. This includes volume percentage of porosity and grain size. Calculated volume percentage porosities matched with

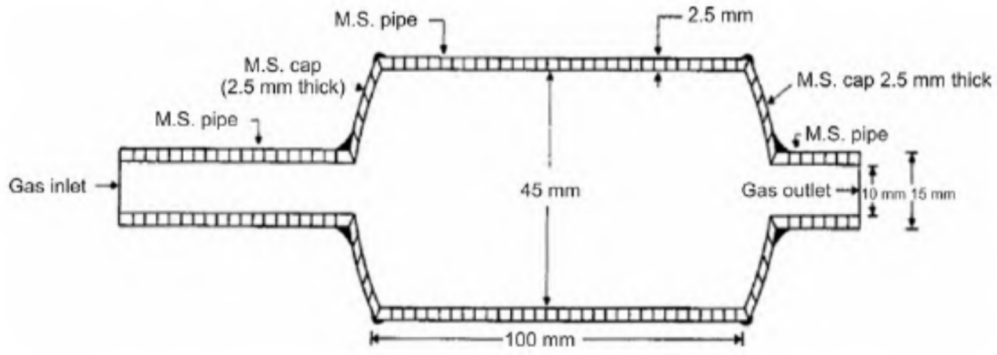

Figure 1. Cross-section of mild steel capsule used in the study.

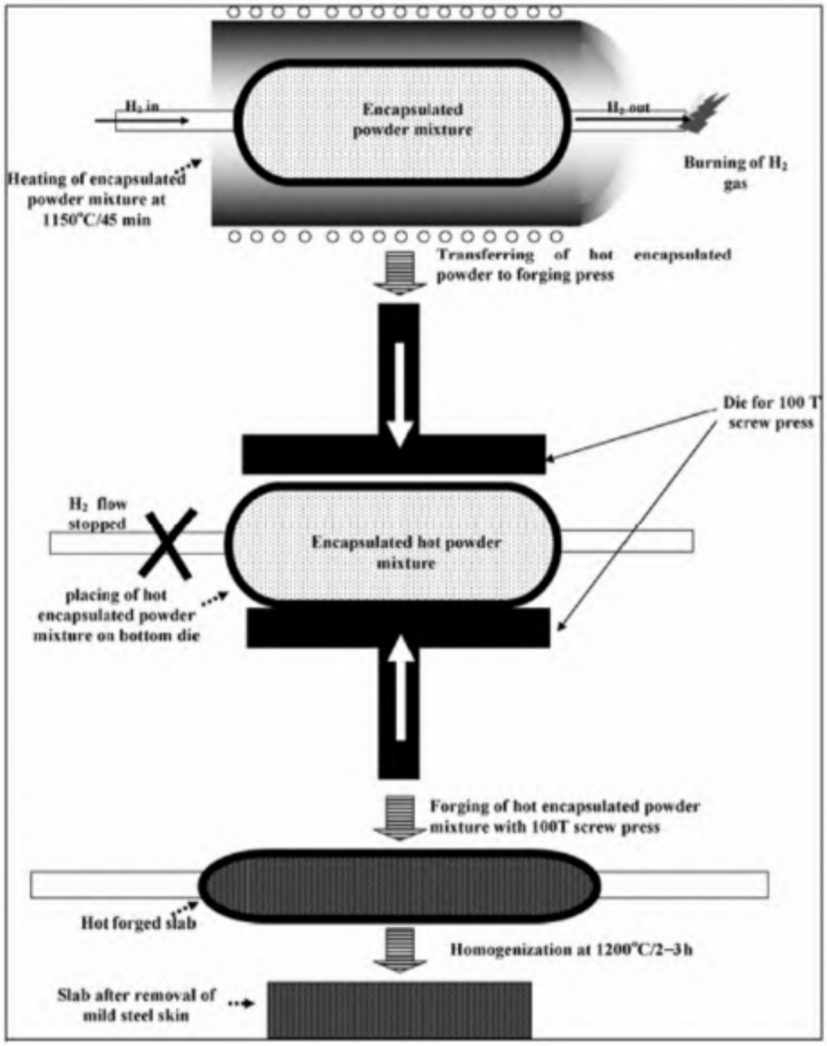

Figure 2. Schematic diagram illustrating the production of slab by hot forging of encapsulated powder mixture.

Table 1. Calculated volume percentage of porosities of the alloys

Sample	Material	As forged density (g/cc)	Rolled and annealed density (g/cc)	Theoretical density (g/cc)	Porosity in rolled and annealed wires, calculated using measured density (vol%)	Porosity in rolled and annealed wires, calculated using quantitative metallographic technique (vol%)
a	Fe–0.35 P	7.27	7.62	7.84	2.8	2.8
b	Fe–0.35 P, 2 Cu, 2Ni,1 Si, 0.5 Mo	7.17	7.58	7.65	1.0	1.7
c	Fe–0.35 P, 2 Cu, 2Ni, 1 Si, 0.5 Mo, 0.15 C	7.50	7.54	7.62	1.0	1.8

the volume percentage porosities measured by the metallographic method. Hardness of the hot rolled and annealed wires were measured with Vicker's hardness tester using 10 kgf load. Samples for tensile testing were either punched out of sheet or wires. The tensile specimens were tested using Hounsfield tensile tester. The tensile testing was carried out at room temperature with a cross head speed of 1 mm/min. Gauge length of the specimens was 20 mm. Gauge diameter of the tensile sample (wires) was 1 mm.

Volume percentage porosities were estimated from the measured density of the specimens. These estimated volume percentage of porosities are recorded in Table 1. In order to verify the correctness of the estimated volume percentage of porosity, the porosities were also measured using quantitative metallographic technique. Rolled and annealed microstructures with the experimentally measured volume percentage of porosity were recorded and shown in Figure 3. They are more or less matching with the theoretically calculated volume percentage porosity. The cross-section of the wires showed almost rounded porosity. Cross-sections of wires were etched to reveal grain boundaries and are shown in Figure 4.

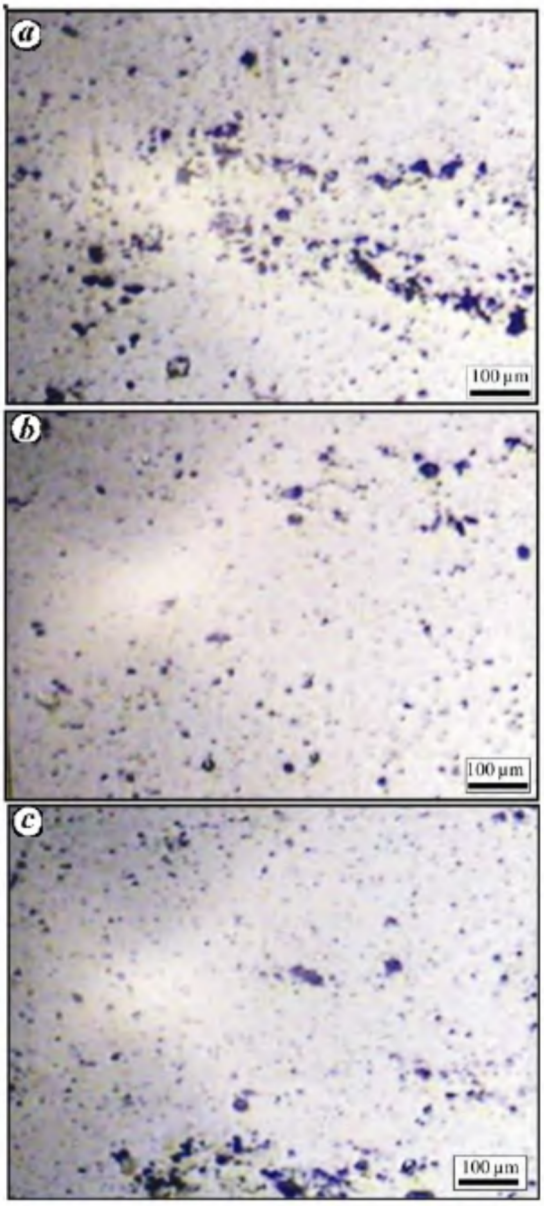

Figure 3. Porosity distribution of the rolled and annealed wires as polished and unetched condition.

In general, samples containing silicon, molybdenum and nickel showed large grains. This may be due to high homogenizing time and temperature used for facilitating effective silicon diffusion. The hardness was found to increase with phosphorus, molybdenum and nickel as well as silicon alloying additions. Figure 5 shows the hardness of different P/M alloys made in this investigation. However, porosity also affected the hardness of these products. The alloy Fe–0.35P with 2.83 vol% porosity showed the hardness of 182 Hv/10 kg. The alloy Fe–0.35P–2Ni–

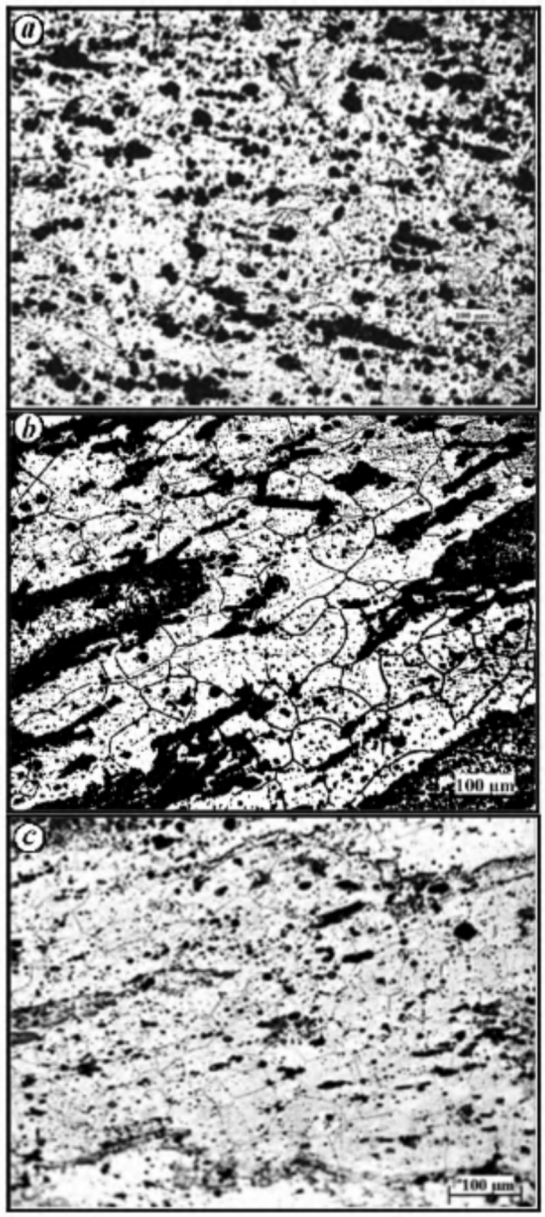

Figure 4. Cross-section of hot rolled and annealed alloys (etched with 2% nital) revealing grain structure. Residual alignment of porosity and fattering of pores are observed in all these rolled and annealed alloys.

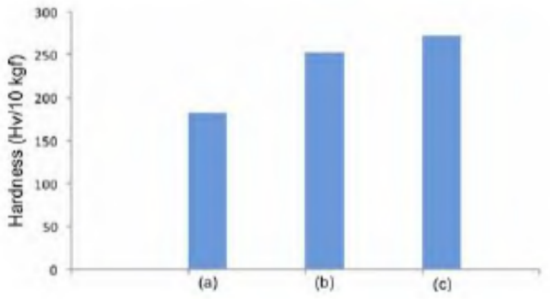

Figure 5. Variation in hardness of P/M alloys (a), (b) and (c).

Table 2. Tensile properties of P/M alloys in fully annealed condition

Sample	Proof stress (MPa)	UTS (MPa)	Total elongation (%)
a	171	296	13
b	282	663	12
c	290	675	14

2Cu–1Si–0.5Mo having 1 vol% porosity and Fe–0.35P–2Ni–2Cu–1Si–0.5Mo–0.15C with 1.03 vol% porosity showed considerable improvement in hardness. Their hardness values are 253 and 272 Hv/10 kg respectively. Had there been similar porosity levels in both of these alloys, improvement in hardness due to phosphorus, molybdenum and nickel alloying addition could have been ascertained.

All the Fe–P alloys showed similar level of ductility. However, the Fe–P based alloys containing other alloying elements (Mo, Ni, Si, C and Cu) show fairly high strength as compared to the one containing 0.35 wt% P. Tensile properties, such as proof strength, tensile strength, percentage elongation of these alloys are shown in Table 2. The mechanical properties obtained in the present investigation are suitable for structural applications. Few limited tensile tests under cold deformed conditions exhibited UTS well over 900 MPa. However ductility came lower marginally. This is possible on account of development of finer grain grain structures due to cold working.

The following conclusions can be drawn from the present investigation are: (i) alloys developed in the present investigation have very good hot workability. (ii) High strength and high ductility are observed in case of alloys containing 0.35 wt% phosphorous. (iii) Alloys containing Mo, Ni, Cu and Si (with or without carbon) showed higher strength (>600 MPa) and higher resilience value with moderate ductility under annealed conditions with scope for developing higher strengths by cold working. (iv) Forged and homogenized as well as rolled and annealed Fe–P based alloys developed in the present investigation were characterized using metallographic technique. All the microstructures showed single-phase ferrite grains with porosities well distributed along the grain boundaries as well as inside the grains. (v) Improvement in hardness levels due to the combined addition of molybdenum, nickel silicon, copper, carbon and phosphorus was found to be comparatively better than that of binary alloys. (vi) Copper significantly reduces the porosity. Binary alloy (Fe–0.35P) shows higher levels of porosity than other two alloys developed in the present study.

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Received 9 February 2009; revised accepted 13 October 2009

Phenotypic diversity of sickle cell disorders with special emphasis on public health genetics in India

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Human genetic diversity poses a great challenge to community health care in India. Haemoglobin disorders constitute the most common genetic and public health burden on vulnerable people. Prospective studies which are lacking in India present valuable community health and morbidity information for analysis with respect to introspection and evaluation. The present study is designed to fill up this lacuna in presenting community health and morbidity pattern of encountered different sickle cell phenotypes in India. 137

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