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Original Research Paper

Reduction and densification characteristics of iron oxide metallic waste during solid state recycling

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ABSTRACT

Powder technology can be used for solid state recycling of metallic waste (e.g. iron oxide as forging scales in the forge shop and grinding sludge from the machine shop). Whereas recycling of such shop floor waste by melting and landfill are well known, that by powder metallurgical process has not received significant attention in published literature. The present work attempts to fill this gap. Initially, waste material (forging scale and grinding sludge) was separately pulverised using ball mill to obtain the respective powders. For carbothermic reduction, graphite (powder of particle size <5 µm) was homogeneously mixed with the two oxide powders. Each of the two mixtures was compacted in a die into a number of pellets. These were sintered for in-situ reduction of the metallic waste. The effect of the source of powder (grinding sludge or oxide scale), quantity of reducing agent cum lubricant, i.e., graphite powder, compaction parameters such as compaction pressure, compact weight and sintering parameters such as sintering temperature and time on reduction and densification characteristics were analysed using Taguchi based experimental design. The results showed significant effect of graphite content and sintering temperature on the degree of reduction and densification. The source of scrap was found to be important since samples from grinding sludge showed higher reduction and densification than forging scale. The analysis of variance was used to establish the individual effects and interactions like that between source of scrap and graphite addition. This was found to contribute most towards degree of reduction and densification. It was found that degree of reduction alone does not necessarily guarantee high degree of densification.

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1. Introduction

Metallic scrap (metallic industrial waste) may be converted into useful products. Physical as well as chemical transformations during recycling of ferrous scrap enable obtaining product properties comparable to those when virgin sources are used.

Over 400 million tonnes of metallic scrap is routinely recycled across the world every year [1,2]. Energy savings in processing material from secondary sources are enormous [3]. Conventionally, scrap is recycled by melting. Recycling metallic scrap begins with physical separation (e.g. magnetic separation, air elutriation, eddy current separation or those involving spectrographic techniques). Subsequent refining includes fluxing, segregation, distillation, and electrolytic techniques [4].

Powder technology permits simultaneous reduction of ferrous scrap (Fe oxide rich) powder and its consolidation during sintering by in-situ reduction of the oxide. Pulverising scrap by ball milling leads to flaky and irregular morphology of particles. Moreover, effectiveness of ball milling reduces with a decrease in the particle size [5,6]. Powder from gas atomisation of molten metal on the other hand would give spherical particles. Better mechanical interlocking of irregular shaped and flaky powder particles is known to make stronger and denser green compacts than those from spherical particles [7].

Materials like ultra-high-strength dual phase steels (which have low carbon content and alloying elements, such as chromium, vanadium, tungsten or molybdenum) have been recycled using powder metallurgy. In these studies, compacting pressure of about 415 MPa was found to give good green strength as well as tensile strength of about 320 MPa and hardness of 280 HV was achieved after sintering at 1075 °C for 60 min [8]. Most of the published literature on recycling of ferrous waste through powder technology pertains to powder obtained through reduction of mill scales which is compacted to PM parts later [9–11].

The present work on the other hand is based on in-situ carbothermic reduction of iron oxide, wherein the iron oxide (with the additives) is compacted to the desired shape and the process of





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reduction occurs during sintering. It is green in the sense that no environmental oxygen is consumed, and reduction of oxide occurs without melting, thereby saving on energy and hence the carbon footprint. Most artificial processes (including the 'environment friendly' ones) consume oxygen, none generate oxygen. The process can still be made more 'green' by using reducing atmosphere consisting of hydrogen or cracked ammonia (which would generate moisture on reducing iron oxide). This will be the subject of a subsequent paper.

The present work therefore aims at studying the in-situ carbothermic reduction of iron oxide aimed at making a powder metallurgical product from ferrous shop floor scrap. An additional objective is to investigate the effect of material and process parameters on reduction and densification characteristics. The process of reduction is briefly explained in the following section.

1.1. Carbothermic reduction of iron oxide

Reduction of iron oxide involves stepwise reactions with reducing agents during which number of atoms of oxygen per atom of iron undergoes significant changes while transforming from stable oxides (Fe₂O₃, Fe₃O₄ and FeO) to pure iron. The sequential reduction of Hematite (Fe₂O₃) \rightarrow Magnetite (Fe₃O₄) \rightarrow Wustite (FeO) \rightarrow Pure Iron (Fe) takes place in two ways, namely, direct and indirect reduction.

In indirect carbothermic reduction, carbon monoxide is formed, which reduces the iron oxide, while in direct reduction, solid carbon (coke, graphite, coal, char, deposited carbon, etc.) comes in contact with iron oxide [12].

$$\begin{aligned} \text{Direct reduction} &: \text{Fe}_x O_y \left(\text{solid} \right) + C \left(\text{solid} \right) \\ &= \text{Fe}_x O_{y-l} \left(\text{solid} \right) + \text{CO} \left(\text{gas} \right) \end{aligned} \tag{1}$$

Indirect reduction : $Fe_xO_v(solid) + CO(gas)$

$$= Fe_x O_{y-l}(solid) + CO_2(gas)$$
(2)

where *x* = 1, 2 or 3 and *y* = 1, 2 or 3.

For Fe₃O₄, carbon monoxide is more efficient than solid carbon as a reducing agent for transformation into pure iron at temperature below 700 °C. Reduction of Fe₂O₃ into Fe₃O₄ and FeO can be carried out by the use of controlled gas mixtures of CO and CO₂. The surface reaction rate is expected to decrease in proportion to the degree of dilution when inert gases such as Ar or N₂ are used with CO. The process starts with direct reduction where carbon (graphite) particles are in contact with iron oxide particles. During this phase CO is produced, and particularly above 700 °C, the Boudouard reaction between formed CO₂ gas and C enhances the quantity of CO which is available for indirect reduction [13]. In the present context, graphite is used for carbothermic reduction of metallic waste and the experimental plan is in accordance with these well known facts.

1.2. Experimental plan

Ferrous scrap was pulverised using laboratory ball mill. Graphite powder is mixed with powdered scrap to ensure even distribution of graphite particles and contact with oxide powder particles on compaction. This mixture was then compacted into a cylindrical compact. During sintering iron oxide reacts with carbon and gets reduced to metallic iron, which also gets densified during sintering. The densification would then be expected to depend upon the extent of reduction of iron oxide, and completeness of this reaction. The detailed experimental plan to approach optimal conditions for densification is shown in Fig. 1. Sintered samples are then characterised for weight and volume change to investigate in-situ reduction and densification processes for PM based recycling technique.

2. Materials and experimental details

2.1. Materials

Forging scale was obtained from a forge shop while grinding sludge came from the machine shop (Fig. 2a and b). The latter contains, besides oxides of iron, filter paper sludge, water and oil.

Forging scales (as shown in Fig. 2(a)) were first crushed and further pulverised using ball milling. Grinding sludge (as shown in Fig. 2(b)) has about 48 wt.% metallic and 52 wt.% non-metallic constituents (of which 86 wt.% was water, 14 wt.% filter paper and the oily sludge) as obtained from TGA data. In order to remove nonmetallic constituents, grinding sludge was dried in still air for 24 h and heated at 350 °C for 30 min in a muffle furnace [14]. Powder from grinding sludge was pulverised using ball milling. Powder obtained from forging scale and grinding sludge was sieved using 100 mesh sieves in order to limit powder particle size to below 150 µm. SEM–EDS study of powder sample was performed to find out the elemental distribution and morphological analysis of powder. The powder was first coated with ultra-thin film of gold by ion sputtering (Anatech, Model: Hummer VI-A) and examined under a Scanning electron microscope (Hitachi, Model: S-3400N). Fig. 3 shows the semi-quantitative abundance (by EDS analysis) of major and minor elements at different locations in the powder sample. These results enable reveal unknown elemental abundance.



Fig. 1. Schematic for powder metallurgy based recycling method.

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