Processing of a High-Alloy High-Speed Steel Via Water Atomisation and Direct Sintering

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Abstract

The common powder metallurgical production route for high-alloy high-speed steels is based on gas-atomised powder, subsequently encapsulated and hot isostatically pressed at temperatures near 1150°C. The billets are further rolled into bars or rods. The microstructures of these materials show very fine carbide distributions which yield high fracture strength values. This paper presents the results of a study of the processing of one such alloy (in wt-%: 2C-4Cr-12W-10Mo-4.5V-12Co, bal. Fe) by means of an alternative near net shape P/M route, using vacuum sintering of die compacted water atomised powder. After heat treatment, this particular steel, produced by this more economical route, has fracture strength values of ~1900 MPa, at a hardness of above 1000 HV30, compared with 2750 MPa for commercial HIPped material of similar composition.

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Introduction

A major advantage of the powder metallurgy (P/M) of high-speed steels (HSS) involving the hot isostatic pressing (HIP) processing route, is the production of fine-grained microstructures without the carbide segregation characteristic of their wrought counterparts, which limits their strength, size stability during heat treatment and grindability. High-alloy grades are only satisfactorily produced by P/M, currently by HIPping gas-atomised powders, whereas conventional grades such as AISI M3/2, T15 and T42 are processed by the more economical route consisting of direct sintering of water atomised powders. Examples of the former route include the Anti-Segregation Process (ASP), developed in Sweden (also known as the *Asea-Stora* process [1]), the American *Crucible* process [2] and the processing of HAP alloys by Japan's *Hitachi* [3]. Consolidation is reported to take place at 80...100°C below the solidus temperature of the alloy and thus these techniques produce very fine microstructures including carbide distributions below 4 µm. Excellent combinations of toughness and wear resistance are reported and, since 1972, the typical transverse rupture strength, s_R , has increased from below 2000 MPa to above 3000 MPa in ASP alloys [1]. The strength-limiting factor has been associated with the size of the fracture-initiating defects and they are currently considered to be oxide inclusions. Previous work [4] by the authors on ASP60 recorded bend strengths above 4000 MPa as well as failure-initiating sites associated with inclusion particles containing aluminium and silicon.

This supersolidus sintering technique employing water atomised powders is directed mainly towards competition with established wrought HSS grades and also provides uniform non-segregated microstructures, but with coarser carbide distributions (up to 30 µm) than those in the HIPped alloys, due to the significantly higher processing temperatures involved. However, production of near net shapes such as cutting tool inserts and engineering components [5,6], yields considerable material and labour savings.

Lately, the Japanese P/M industry has also developed "tough and hard" high-alloy high-speed steels with superior cutting and wear-resistant properties. One such alloy is Hitachi's HAP 70 (2C-4Cr-12W-10Mo-4.5V-12Co-bal. Fe, in wt-%) [3], with a fine microstructure, including small round carbides. For such alloys, it is important to ascertain to what extent mechanical properties are associated with chemical composition, processing and/or details of microstructure, respectively. Accordingly, at the suggestion of *Sintermetallwerk Krebsöge* (Germany), it was decided to process an alloy of similar chemical composition via the water atomisation plus vacuum sintering route and compare the resulting microstructures and mechanical properties with those of the commercially available alloy.

Experimental

The atomisation of the experimental alloy was performed in a water atomiser with 5 kg capacity. The melt was poured directly in the atomiser tundish where it passed through a calibrated hole (4.1 mm diameter) located at the bottom of the crucible, then desintegrating due to the high-pressure (20 MPa) water jets coming from the nozzles. In order to minimise the oxidation of the powder during the atomisation process, the atmosphere inside the atomiser was flowing argon. The temperature of the melt in the atomiser tundish was determined using a Pt-Pt 13% Rh thermocouple placed inside the crucible and protected with a silica tube.

The powder decanted at the bottom of the atomiser was filtered and dried under vacuum (~1.3 10^{-4} MPa) at 80°C for 24 h. The dried powder was then sieved in order to separate the fraction < 125 µm, which was subsequently vacuum annealed in a pilot-scale furnace at 950°C for 3 h to soften and deoxidise the material.

Two fractions of the annealed powder were blended with graphite (0.1 and 0.2 wt-%) in a Turbula mixer for 2 h. The powders were then unidirectionally compacted to specimen shape having the approximate dimensions of 20x6x4 mm³. The sintering curves were determined by implementing the following sintering cycle: heating up to the sintering temperature at 10K/min, with a hold of 15 min at 1000°C for degassing; furnace cooling the samples to room temperature after 60 min at the sintering temperature. The densities of the sintered specimens were determined by the Archimedes method, the specimens having been previously coated with lacquer in order to seal the surface against water penetration.

The sintered samples were hardened and tempered. The hardening treatment was carried out in salt baths: the specimens were pretreated at 800°C for 5 min in a salt bath, then

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Table 1 Chemical composition of the experimental powder after annealing [wt-%, bal Fe]

C	Cr	W	Mo	V	Co	O	N
2.0	4.1	11.4	11.5	4.4	11.8	0.17	0.028

austenitised in another salt bath and finally oil-quenched. The austenitisation soaking time, which was in fact the time of immersion of the specimens in the high-temperature salt bath, was ~1 min in all cases. The tempering curves were determined by triple tempering for 1 h in a muffle type furnace, the specimens being imbedded in a mixture of alumina and graphite, in order to minimise oxidation.

The mechanical testing involved three-point-bending on a 50kN tester, at a cross-head speed of 0.1 mm/min and a span of 16 mm. The transverse rupture strength (s_R) was calculated using the following relationship, according to the ASTM standard B 528-76:

$$s_R = \frac{3Pl}{2bh^2} \quad (1)$$

where P = load, l = span, b = specimen width and h = specimen depth. The test specimens were ground with diamond wheels on all surfaces to the final dimensions of 20x4x2 mm^3. All grinding was longitudinal and the tensile faces were subsequently metallographically polished to 3 µm diamond paste.

The specimens before and after failure were metallographically examined by optical and scanning electron microscopy. The identification of the phases in the microstructures of both as-sintered and as-heat-treated specimens was complemented by X-ray diffraction analysis.

Results

Properties of the Water Atomised Powder

The mean particle size of the powder was 86 µm and the fractions with a mean particle size below 125 µm corresponded to 81.1 % of the total weight of the powder. The low value of the apparent density (1.91 $g \cdot cm^{-3}$) was due to the very irregular shape of the particles, but a reasonable flowability (53 s/50 g) was found to be the case for the powder. The as-atomised powder had a dendritic solidification microstructure which, after annealing, was transformed into a dispersion of spheroidised carbides in a ferritic matrix. The annealing treatment under vacuum at 950°C resulted in the oxygen content being lowered from 0.24 to 0.17 %, with a corresponding drop in carbon level from 2.11 to 2.0 % (see Table 1 for a complete analysis).

The compressibility tests of the < 125 µm fraction of the annealed powder were carried out between 200 and 900 MPa, which is the usual compacting pressure range for annealed high-speed steel powders. Green densities of about 70 % (taking the density of the as-cast material as a reference) were obtained at 600 MPa. Following previous experience [4,7], the compacting pressure used in this work was 830 MPa.

Sintering

The solidus temperature, determined by F. Lemoisson and Y. Bienvenu, using DTA during the heating stage, was found to be 1223°C [8]. This implies that the plateau of the sintering curve is expected to start at this temperature, according to the supersolidus liquid phase sintering models proposed by German [9] and Wright [10]. The sintering curves of the powder, with and without carbon additions, were determined as being within the range of 1180...1250°C (Fig. 1). The sintering plateau was found to start at 1230°C for the powder with no carbon additions, and at 1220 and 1210°C in the case of 0.1 % and 0.2% carbon additions, respectively.

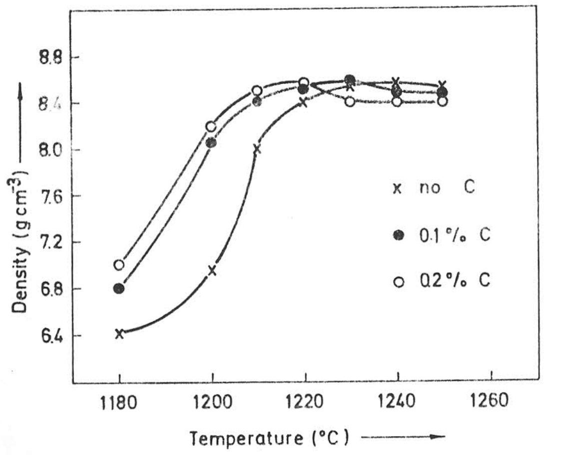

Fig. 1 Effect of carbon additions on the sintering behaviour of the annealed experimental powder

The microstructures obtained at the start of the sintering plateau in each case are shown in Fig. 2, and they are substantially different from the microstructure of the commercial alloy regarding size and morphology of the carbides. The microstructure of HAP 70 [3] is characterized by a fine (2...3 µm) distribution of rounded carbides. In the vacuum-sintered alloy of similar composition processed at higher temperatures, angular M_6C and rounded MC-type carbides were dispersed in a predominantly bainitic matrix (as a result of the rather fast cooling rate in the sintering furnace), yielding hardness values of 700...720 HV30. Needle-like M_2C carbides were observed in some samples with carbon additions. The identification of the above phases was confirmed by X-ray diffraction analysis, which also revealed some retained austenite. Slightly oversintered microstructures were observed at 1250°C and at 1240°C for the powder with and without carbon additions, respectively. This means that the optimum sintering temperature range covers less than 20°C.

Although the carbon additions caused a 10...20°C decrease in the sintering temperature of the experimental powder, all further studies were carried out using the powder with no carbon addition in order to avoid the formation of carbides deleterious to the mechanical properties. The sintering temperature used was 1240°C, which falls within the optimum sintering temperature range.

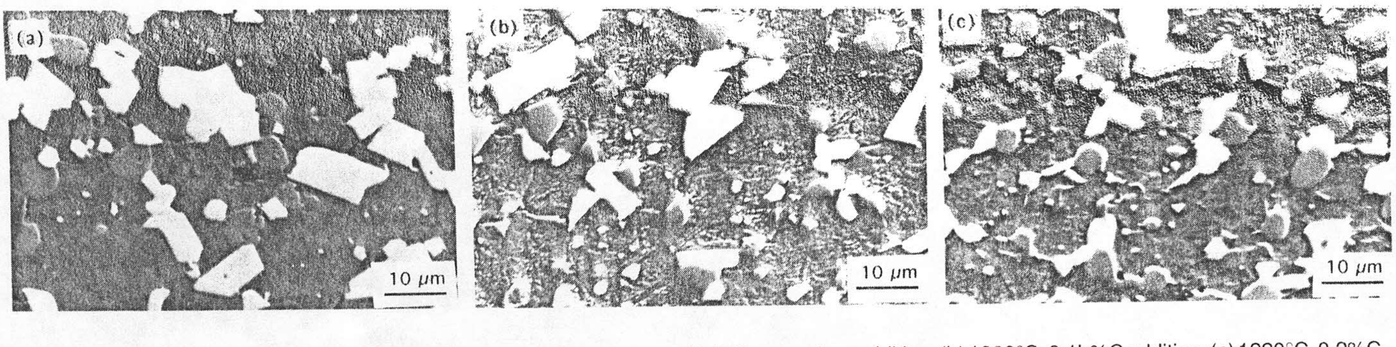

Fig. 2 As-sintered microstructures of the experimental alloy sintered at: (a) 1240°C, no carbon addition; (b) 1230°C, 0.1b%C addition; (c) 1220°C, 0.2%C addition

therefore supports the supersolidus liquid phase sintering model proposed by German [9] and Wright [10].

In accordance with the literature [1,2], the main difference between the microstructures of the commercial HIPped and laboratory vacuum-sintered high-alloy steels is carbide size, which is a direct consequence of the processing temperatures: below the solidus (probably close to 1150°C) for the commercial powders and above the solidus (1240°C) for the water atomised powders. HIPping also promotes greater microstructural integrity, such that failure initiation is associated with inclusions [4] rather than carbides or weak interfaces [11]. Accordingly, HIPped high-speed steels generally possess a higher bend strength, but not necessarily toughness [12]. The bend strengths reported in the literature [3] for the HAP 70 steel were 2750 MPa for a 71.6 HRC (~1000 HV), compared to ~1800 MPa for the material under discussion, which is of the same magnitude (2000 MPa) as a T15 steel processed via the same route [7]. It is suggested that the angular and coarse M_6C carbides of the experimental alloy may be responsible for the initiation of the cracks, due to both their size and angular shape (Fig. 5). These cracks can then, perhaps subcritically at first, propagate through the matrix, either transgranularly or following weak interfaces associated with e.g. previous particle boundaries or previous austenite grain boudaries with carbide films.

Conclusions

This work shows that the high-alloy high-speed steel experimental powder with the composition 2C-4Cr-12W-7Mo-4.5V-12Co-bal. Fe (in wt-%) can be successfully water atomised and vacuum sintered as near net shapes. The sintering temperatures (1230...1250°C) cover the lower range of the usual sintering temperatures of water atomised and annealed high-speed steel powders. The fracture strength values obtained in bending tests (~1800 MPa) were similar to those reported for other high-speed steels processed by this route.

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Kurzfassung - Résumé

Herstellung eines hochlegierten Schnell-Arbeitsstahls durch Wasserverdüsung und direktes Sintern

Die übliche pulvermetallurgische Herstellungsmethode für hochlegierte Schnell-Arbeitsstähle beruht auf gasverdüstem Pulver, das eingekapselt bei Temperaturen nahe 1150°C heißisostatisch gepreßt wird. Die Brammen werden weiter in Blöcke und Stangen umgeformt. Die Gefüge dieser Materialien zeigen sehr feine Karbidverteilungen, die hohe Bruchfestigkeit ergeben. In diesem Aufsatz werden Ergebnisse einer Studie vorgestellt, in der eine solche Legierung (in Gew-%: 2C-4Cr-12W-10Mo-4,5V-12Co, Rest Fe) mit einer alternativen Nahe-Endform PM-Route hergestellt wurde, die auf dem Vakuum-Sintern verpreßter, wasserverdüster Pulver beruht. Schnell-Arbeitsstahl, der mit dieser wirtschaftlicheren Methode hergestellt wurde, zeigte nach der Wärmebehandlung Bruchfestigkeiten von etwa 1900 MPa bei Härten oberhalb 1000 HV30 im Vergleich zu Festigkeiten von 2750 MPa, wie sie kommerziell erhältliche geHIPte Materialien ähnlicher Zusammensetzung zeigen.

Fabrication d'acier rapide hautement allié par atomisation à eau et frittage direct

Le procédé classique de fabrication d'cier rapide hautement allié est basé sur de la poudre atomisée en phase gazeuse, qui est par la suite encapsulée et pressée isostatiquement à chaud à des températures d'environ 1150°C. Les brammes sont ensuite enroulées en barres ou toubes. Les microstructures de ces matériaux présentent de trés fines distributions des carbides, qui conduisent à une haute résistance à la fracture. Cet article présente les résultats d'une étude d'un tel alliage (% en masse: 2C-4Cr-12W-10Mo-4.5V-12Co, reste Fe) obtenu par une autre méthode P/M avec forme finale approchée, utilisant le frittagge sous vide de compactes de poudre atomisée par eau. Aprés traitement thermique, cet acier particulier fabrique selon cette méthode plus économique a des valeures de résistance à la fracture d'environ 1900 MPa pour une dureté supérieure à 1000 HV30, en comparaison des 2750 MPa des matériaux commerciales pressés isostatiquement à chaud et d'une composition similaire. ❑

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Powder Metallurgy - an Overview

I. Jenkins, J.V. Wood (Eds.)
The Institute of Metals, London, 1991, 320 pp., 21 cm x 27 cm, hardcover, US-$ 110,- (English)

As the first volume of a new *Institute of Metals* series on powder metallurgy, this reference work provides authoritative and critical reviews by renowned international experts regarding the important advances which are being made in P/M science and practice. The specific contributions cover a broad spectrum of processes etc. related to powder metallurgy, ranging from powder production through consolidation, sintering, applications, design and testing. The contents are subdivided into three chapters based on five thematic areas:

1 Powder Production (atomization, spray casting, production and handling of electrolytic powders, spark erosion)

2 Powder Characterization (size and morphology, surface chemical characterization of powders)

3 Consolidation (metal injection molding, cold isostatic pressing, dynamic compaction, rotary forging as well as sintering and HIP diagrams)

4 Sintering (solid state and liquid phase sintering, sintering aids in P/M, selection of sintering atmospheres)

5 Applications (light metals, composites, ferrous materials, HSS, wear- and corrosion-resistant coatings, cemented carbides, refractory metals, P/M materials for electrical engineering, magnetic powders, superconductors, biomaterials, quality assurance).

This book is primarily directed towards meeting the needs of undergraduates by presenting the basic scientific and technological concepts of powder metallurgy, supported by appropriate examples of industrial practice. It will be of value to research workers at institutes and in industry, and to practising powder metallurgists and design engineers as a general textbook.
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