Materials Science and Engineering A 499 (2009) 360–367

Contents lists available at ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

# Microstructural characterisation of vacuum sintered T42 powder metallurgy high-speed steel after heat treatments

V. Trabadelo*, S. Giménez[1], I. Iturriza

*CEIT and TECNUN, Paseo Manuel de Lardizabal 15, 20018 San Sebastián, Spain*

ARTICLE INFO

*Article history:*
Received 17 March 2008
Received in revised form 30 July 2008
Accepted 27 August 2008

*Keywords:*
High-speed steel
Tempering
Annealing
Secondary precipitation
Transmission electron microscopy

ABSTRACT

High-speed steel powders (T42 grade) have been uniaxially cold-pressed and vacuum sintered to full density. Subsequently, the material was heat treated following an austenitising + quenching + multitempering route or alternatively austenitising + isothermal annealing. The isothermal annealing route was designed in order to attain a hardness value of ~50 Rockwell C (HRC) (adequate for structural applications) while the multitempering parameters were selected to obtain this value and also the maximum hardening of the material (~66 HRC). Microstructural characterisation has been carried out by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The microstructure consists of a ferrous (martensitic or ferritic) matrix with a distribution of second phase particles corresponding to nanometric and submicrometric secondary carbides precipitated during heat treatment together with primary carbides. The identification of those secondary precipitates (mainly $M_3C$, $M_6C$ and $M_{23}C_6$ carbides) has allowed understanding the microstructural evolution of T42 high-speed steel under different processing conditions.



## 1. Introduction

High-speed steels (HSSs) have been traditionally used as cutting tools and wear parts, applications where a high hardness (60–70 Rockwell C (HRC)) and wear resistance are needed [1]. This imposes the application of quenching and tempering as the final heat treatment. More recently, these materials have also been employed for structural purposes, such as high performance exhaust valve seat inserts (VSIs) [2]. In fact, there are now several commercial solutions based on HSSs for VSI trying to encompass a good wear resistance at service temperature with microstructural stability, good machinability, low cost and robust processing [3,4]. In general terms, these structural applications require a combination of high strength, wear resistance and hardness together with a moderate toughness and fatigue resistance. Intermediate hardness values between 35 and 55 HRC have proven to be adequate for such applications [5]. These hardness values can be obtained by different heat treatments like isothermal annealing at temperatures far below $A_1$, overtempering or transformation annealing [6–8]. In any case, heat treatments are necessary to tune the mechanical properties of HSSs to the final requirements and consequently, the effect of the heat treatments on their microstructure and mechanical properties has constituted a very dynamic research field during the last years. In this context, considerable efforts have been focused on the microstructural changes occurring during tempering. It is well known that after tempering at 520–550 °C, the so-called secondary precipitation of HSSs is responsible for the increased hardness values obtained. The precipitates consist of two main types of nanometric size carbides: MC and $M_2C$. When tempering is carried out at temperatures around 600–800 °C (overtempering), MC and $M_2C$ coalesce until they finally disappear and are substituted by the more stable $M_6C$ and $M_{23}C_6$ carbides [9–14].

In the present study, the effect of both multitempering and isothermal annealing on the microstructure of the T42 powder metallurgy HSS is analysed. The main objective is to understand the microstructural evolution of this material leading to the desired mechanical properties. Hardness has been chosen as a parameter representative of the mechanical behaviour of this material, since hardness is related to toughness [15] and therefore to ductility. In that sense, 50 HRC has been targeted for structural applications. A specific study on the mechanical properties (fracture toughness and fracture strength) of these microstructures was previously reported [16]. Additionally, when the same hardness is obtained by different heat treatments, the microstructural correlation has been studied.

* Corresponding author. Present address: Tekniker, Avda. Otaola 20, 20600 Eibar, Spain. Fax: +34 943202757.
*E-mail address:* vtrabadelo@tekniker.es (V. Trabadelo).
[1] Present address: Departamento de Ciencias Experimentales, University Jaime I, 12071 Castellón de la Plana, Spain.

0921-5093/$ – see front matter © 2008 Elsevier B.V. All rights reserved.
doi:10.1016/j.msea.2008.08.043

**Table 1**
Chemical composition of the as-received T42 HSS powders (weight percentage except for *ppm).

| | |
|---|---|
| C | 1.32 |
| Co | 10.58 |
| Cr | 4.20 |
| Mo | 3.82 |
| O* | 603 |
| Si | 0.31 |
| V | 3.32 |
| W | 9.98 |
| Fe | Bal. |

## 2. Materials and experimental method

The hydrogen annealed water atomised T42 powder grade was supplied by Höganäs Great Britain Ltd. (Aylesbury, United Kingdom). The composition of the material is given in Table 1. The particle size distribution measured by the manufacturer is given in Table 2. The mean particle size calculated from Table 2 is 69 μm. A 0.6 wt% compaction lubricant and a 0.4 wt% carbon in the form of graphite powder (<5 μm) were added to improve both the compressibility and the sinterability of the alloy, respectively. The powders were blended in a TURBULA© device (Glen Mills Inc., Clifton, NJ) during 2 h at a constant speed (30 rpm). The microstructural characterisation of the powders has been reported elsewhere [17]. Subsequently, the powders were uniaxially cold-pressed at 600 MPa to 16 mm diameter cylinders with a density of 5.75 g/cm$^3$ (approximately 70% theoretical density). Full densification of the cold-pressed green compacts was achieved by vacuum sintering ($P = 10^{-1}$ mbar). The samples were heated up at 10 °C/min and kept at 450 °C for 15 min for the complete elimination of the compaction lubricant. Subsequently, they were heated up to the sintering temperature at 10 °C/min, soaked for 60 min and cooled down to room temperature inside the furnace (average cooling rate <10 °C/min). The sintering temperatures have been varied from 1180 to 1250 °C.

The as-sintered compacts were austenitised for 15 min in an Ar atmosphere and then followed either:

(a) Oil quenching + triple 1 h tempering in an air muffle-type furnace (intermediate cooling segments were carried out under air). The austenitising temperature selected was 1000 °C. Tempering temperatures varied from 475 to 650 °C.
(b) Isothermal annealing in a salt furnace where temperature varied between 600 and 675 °C and time changed from 3 to 24 h. The austenitising temperatures selected were 1000, 975 and 950 °C.

Densities were measured by the Archimedes' method. Cross-sections were mechanically polished, etched with 2% nital and observed by scanning electronic microscopy using a Philips XL30-SEM (Eindhoven, The Netherlands) equipped with an EDAX (Tilburg, The Netherlands) energy dispersive X-ray spectroscopy system (EDS). The mean grain size, carbide volume fraction ($f_v$) and carbide mean size (characterised by the mean equivalent diameter, $d_{eq}$) were measured by quantitative metallography techniques (LEICA Qwin image analysis software [18]). The submicron and nanometric precipitates observed after heat treatment were characterised by transmission electron microscopy (TEM). Extraction replicas were obtained by etching the mirror polished cross-section of the specimens with 2% nital or with diluted Villela's reagent and subsequently coating with a thin layer of carbon. The replicas were stripped from the specimens by electrolytic etching in 10% nital using a potential of approximately 30 V. The examination was carried out at 100 kV with a Philips CM-12 TEM ($LaB_6$ filament) fitted with an EDS system. The chemical composition of the extracted precipitates has been determined by analysing at least 30 particles by EDS. Thin foils for the observation of the matrix were prepared by $Ar^+$ ion milling of 3 mm diameter samples in a Gatan model 691 (Pleasanton, CA). The samples were previously mechanically thinned to ~100 μm and both sides were polished. The hardness tests were performed with an ATK-F1000 Mitutoyo hardness tester (Japan). The measurements were carried out at 150 kg and reported in Rockwell C units. The volume fraction of retained austenite was measured by X-ray diffractometry using the method proposed by Maeder et al. [19].

**Table 2**
Particle size distribution of the as-received T42 HSS powders (weight percentage).

| Particle size (μm) | wt% |
|---|---|
| >250 | 0.00 |
| >180 | 0.01 |
| >150 | 0.00 |
| >106 | 10.43 |
| >75 | 22.22 |
| >45 | 47.00 |
| <45 | 20.34 |

**Table 3**
Carbon, nitrogen and oxygen contents for the specimens of T42 + 0.4%C after sintering at the optimum sintering temperature (OST).

| | |
|---|---|
| C (wt%) | 1.60 |
| N (wt%) | 0.01 |
| O (wt%) | 0.021 |
| Density (g/cm$^3$) | 8.19 |
| %T.D. | 99.70 |
| OST (°C) | 1220 |
| SW (°C) | ~10 |

Sintering window (SW) is also reported.

## 3. Results

### 3.1. Sintering behaviour

The optimum sintering temperature (OST) for the material with the 0.4 wt% carbon addition is 1220 °C, and the sintering window (SW) around 10 °C; while for T42 without the extra carbon content the OST is 1260 °C and the SW less than 5 °C. Therefore, it is clear the effect of C reducing the OST and enlarging the SW as previously reported [20]. The sintering data and the C, N and O contents after sintering are listed in Table 3. The microstructure of the material sintered at the OST is shown in Fig. 1. It consists of a dispersion of W and Mo rich $M_6C$ carbides and V rich MC carbides embedded in a pearlitic matrix. The volume fraction ($f_v$), size of carbides ($d_{eq}$) and the mean grain size ($L$) are summarised in Table 4.

**Table 4**
Volume fraction and equivalent diameter of the primary $M_6C$ and MC carbides in T42 + 0.4%C after sintering at 1220 °C.

| | |
|---|---|
| $M_6C$ | |
| $f_v$ (%) | 5.4 ± 0.6 |
| $d_{eq}$ (μm) | 1.39 ± 0.03 |
| MC | |
| $f_v$ (%) | 8.4 ± 0.6 |
| $d_{eq}$ (μm) | 1.63 ± 0.07 |
| Total (%) | 13.8 ± 1.2 |
| $L$ (μm) | 15.0 ± 1.0 |

The mean grain size ($L$) is also included.