



# Improved the microstructures and properties of M3:2 high-speed steel by spray forming and niobium alloying

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## ABSTRACT

The microstructures and properties of spray formed (SF) high-speed steels (HSSs) with or without niobium (Nb) addition were studied. Particular emphasis was placed on the effect of Nb on the solidification microstructures, decomposition of  $M_2C$  carbides, thermal stability and mechanical properties. The results show that spray forming can refine the cell size of eutectic carbides due to the rapid cooling effect during atomization. With Nb addition, further refinement of the eutectic carbides and primary austenite grains are obtained. Moreover, the Nb addition can accelerate the decomposition of  $M_2C$  carbides and increase the thermal stability of high-speed steel, and also can improve the hardness and bending strength with slightly decrease the impact toughness. The high-speed steel made by spray forming and Nb alloying can give a better tool performance compared with powder metallurgy M3:2 and commercial AISI M2 high-speed steels.

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## 1. Introduction

High-speed steels (HSSs), as a special class of highly alloyed tool steels, are extensively used as cutting tools, cold molds and rolls for the exceptional combination of excellent hot hardness, high wear resistance, well toughness and good workability. These properties are mainly depended on the size, type, distribution, shape and amount of carbides and the state of the matrix [1–3].

HSSs made by conventional ingot casting contain coarse network of eutectic carbides formed at grain boundaries. Even after certain forging degree, the carbide particles are arranged in bands parallel to forging direction, which deteriorating the isotropic properties and toughness of HSSs [4,5]. On the other hand, the slow cooling rate of conventional ingot casting limited the addition of strong carbide forming elements such as Ti, Nb, V due to the formation of coarse carbides. Fortunately, the rapid solidification technology provides a new opportunity to modify the solidification microstructures and improve the properties of HSSs, e.g., powder metallurgy (PM) was the first and the most mature industrial application of the rapid solidification technology for high-speed steel products. Finer and more uniform primary carbides, smaller grain sizes and the absence of carbides stringers and macro-segregation are some characteristics attained by PM HSSs compare with the conventional ingot casting HSSs [6–8]. Also it is possible to produce any alloy design by PM process. However, the PM HSSs is still applied in a narrow field because they have normally high cost due to the complex and rigorous processing steps.

Spray forming (SF), a promising preparation technology for materials, can combine the rapid solidification (gas atomization) and near-net-shape forming (deposition) and has been proved to be an effective method to improve the solidification microstructures of tool steels [9–12], due to the high cooling rate ( $10^3$ – $10^5$  K/s) during gas atomization and the microstructural deformation or fracture in deposition stage [13]. In contrast to conventional ingot casting, the most significant microstructure characteristics of SF HSSs are the smaller equiaxed grains, finer and much more uniform-distributed carbides, and less degree of segregation [6,8], resulting in the improved performance of SF HSSs. However, the increase of cooling rate in atomization stage is limited by the density of the final billet [14–16].

Alloying design has been widely used to improve the microstructures and properties of HSSs, and two main alloying strategies are proposed: adding non-carbide forming elements (e.g., Co, Al, Si and rare earth) [17–19], or modifying the microstructures and properties with strong carbide forming elements (e.g., Ti, Nb, Zr). Recently, a relatively great number of literatures reflect the enhanced interest of researchers in various countries on the use of Nb and Ti for alloying HSSs [20–27], but most of the mentioned works were based on conventional ingot casting.

In this paper, the idea of combining spray forming and Nb alloying to improve the performance of M3:2 HSS is implemented and the purpose of the present work is to study the microstructures and mechanical properties of spray formed Nb-containing HSSs as well as the role of Nb in HSSs.

## 2. Experimental procedure

Billet with dimension of  $\Phi 180$  mm  $\times$  100 mm was prepared by spray forming and the processing parameters are shown in Table 1. A

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**Table 1**  
Process parameters of spray forming.

Items	Parameters
Super heat/°C	160–180
Diameter of the delivery tube/mm	4.0
Atomization gas	N <sub>2</sub>
Atomization pressure/MPa	0.45–0.5
Deposition distance/mm	425–450

**Table 2**  
Chemical compositions of the investigated steels (wt.%).

	C	W	Mo	Cr	V	Nb
Steel A	1.30	6.20	5.10	4.60	2.80	–
Steel B	1.31	6.10	4.90	4.48	2.75	0.50

commercial AISI M2 HSS used as the feedstock was melted with appropriate additions of alloying elements. The chemical compositions of the experimental steels are listed in Table 2.

A cylindrical specimen with a size of  $\Phi 50$  mm  $\times$  40 mm was cut from the center of the spray-formed billet and subjected to isothermal annealing at 1180 °C for 1.5 h, then forged to  $\Phi 16$  mm bars at 1150 °C followed with slowly cooling to room temperature. Before the hardening treatment, all the specimens were heated to 900 °C with a heating rate of 100 °C/h and soaked for 1.5 h, then cooled to 25 °C with a cooling rate of 20 °C/h. Subsequently, the specimens were austenitized at 1160–1220 °C for 25 min and oil quenched to room temperature, followed by triple tempering at 540–600 °C for 1 h. In order to prevent decarburization during heat treatment, each sample was sealed in vacuum quartz tube.

The microstructures were characterized using field emission scanning electron microscope (SEM), and Energy dispersive X-ray spectroscopy (EDS) was employed for local chemical composition determination and for the distinction of MC and M<sub>2</sub>C carbides. For metallographic examinations, the samples were prepared by grinding and polishing, and were etched with 8% nital solution. The carbide volume fraction as well as grain size was measured by using image analysis software (Image-pro plus 6). The reported values for each sample were

taken from at least 50 measurements in different fields with a magnification of 1000 times. The extraction of carbides was performed using electrolysis, operating at 40 V, 0 °C with an electrolyte contained 7 g citric acid, 20 mL hydrochloric acid and 250 mL methanol. X-ray scattering was made with a Rigaku X-ray Diffractometer (D/MAX-RB) with CuK $\alpha$  radiation ( $\lambda = 1.5406$  Å) at 12 kW. Differential scanning calorimetry (DSC) experiments were carried out in a Netzsch DSC calorimeter with cooling rate of 10 K/min from 1455 to 200 °C. The DSC samples ( $\Phi 5$  mm  $\times$  1 mm) were machined from the as-deposited billet.

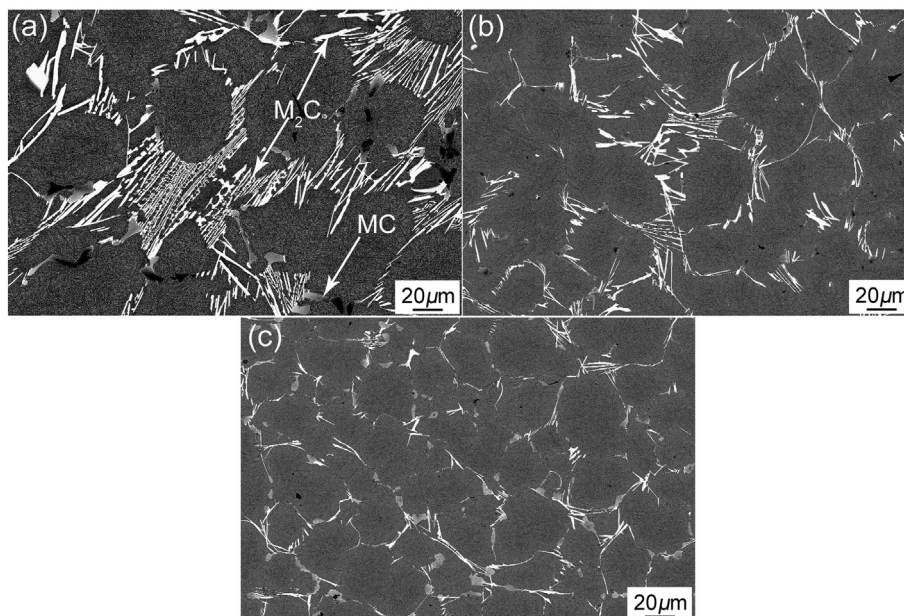
Three-point bending tests were carried out on specimens with dimension of 5  $\times$  5  $\times$  35 mm at a bending rate of 0.1 mm/min. The sample size for impact toughness tests was 10  $\times$  10  $\times$  55 mm without notch according to ISO 5754. The bulk hardness measurements were carried out using Rockwell “C” scale with a load of 150 kgf (589 N) applied for 3 s and 7–10 readings were considered for estimating the hardness values.

The reamers machined from the forged bars were sealed in vacuum quartz tube and annealed at 900 °C for 1.5 h, and then austenitized at 1200 °C for 25 min and oil-quenched to room temperature, followed by triple tempering at 560 °C for 1 h. The reamers used to ream the medium carbon steel with average hardness of 32HRC, and all the tests were under the condition of oil lubrication.

### 3. Results and discussion

#### 3.1. Effects of spray forming and Nb alloying on the microstructures of HSSs

The microstructures of the conventional ingot casting HSS and SF HSS in Fig. 1 show that the solidification microstructures are consisted of equiaxed grains, discontinuous network of plate-shaped M<sub>2</sub>C eutectic carbides (white) and MC carbides (gray) both at grain boundaries and within the matrix. It can be obtained that the grain size of SF HSS is similar to that of conventional ingot casting HSS ( $\sim 60$   $\mu$ m). However, the sizes of M<sub>2</sub>C and MC carbides in SF HSS are remarkably refined (Fig. 1 a and b), e.g., the cell sizes of M<sub>2</sub>C eutectic carbides in conventional ingot casting HSS and SF HSS are  $\sim 35$   $\mu$ m,  $\sim 17$   $\mu$ m, respectively. R.A. Mesquita and C.A. Barbosa [4] reported that high cooling rate of atomization is the main factor for primary carbides refining. Thus, the cooling rate could be the key factor to refine the solidification microstructure of



**Fig. 1.** Microstructures of steel A (a, b) and B (c) made by ingot cast (a) and spray forming (b, c).

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