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# Improving properties of spring steel through nano-particles alloying



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# ABSTRACT

The main challenge in realizing the immense potential of nano-engineered steels is to manufacture large components and quantities at low costs, where homogeneous dispersion of ceramic nano-particles in molten metals is extremely hard to achieve. Therefore, the aim of the present work was to investigate the potential of different nano-particles as alloying elements in the conventional liquid metal casting process and to define their effect on the tensile properties, fracture toughness and fatigue resistance of the spring steel. Results of this investigation indicate that nano-particles reinforcement of spring steel is feasible even by using simple liquid metal casting routes, with the nano-particles type and size having important impact. In the case of larger Al<sub>2</sub>O<sub>3</sub> nano-particles with the highest stability tensile strength of spring steel was improved for up to 10%, while finer and more uniform distribution combined with local reinforcement of the steel matrix in the case of C-nanotubes lead to better fracture toughness and fatigue properties.

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### 1. Introduction

In the last decades, considerable efforts are made in the development of high and even ultra-high performance steels [1,2]. Part of the demand is due to needs for more compact design and cost reduction in the automotive industry and part due to environmental concerns. The environmental aspect is related to reduced CO<sub>2</sub> emission and improved fuel consumption [3]. In this respect, using lighter components and reducing vehicle weight is one of the key elements. However, lighter components require high strength materials where improvement in strength should not degrade other properties such as formability, toughness and fatigue resistance [4]. Moreover, keeping high reliability is the first priority. Growing economic and environmental constraints are affecting the truck industry even more than the passenger-car industry. In the case of trucks redesign and use of lighter high strength leaf springs can bring a big benefit. While increasing design flexibility and making room for additional safety components, it reduces truck weight and related fuel consumption and CO2 emission. Parabolic leaf springs used in suspension systems of truck front axles are usually made of two leaves, and serve two main purposes: support the weight of the trailer and also provide the spring function in the suspension system. With the new spring designs, eventually aiming at a single leaf solution, savings of over 20% of the total weight of the spring can be expected. However, this would also lead to about 10–15% increase in spring maximum stress, which requires better spring steel with ultimate tensile strength of over 2000 MPa [5–7].

In the case of spring steels, the emphasis in materials research is focused on increasing the strength while maintaining good ductility, toughness and fatigue properties [4,7,8]. Over the last decades considerable efforts have been made to develop high strength spring steels to meet the needs for weight and cost savings in the automotive industry [9]. Improved strength of spring steel can be achieved through control of alloy composition, effective heat treatment, micro-alloying, thermo-mechanical treatment and shotpeening [9-13]. The biggest gains in terms of martensitic steel strength improvement are achievable by lowering the austenitizing and tempering temperature, which increase ultimate tensile strength but on the other hand reduce material ductility and toughness [11]. Another way of improving spring steel properties is through grain refinement [10,13,14], Thus many attempts have been made to increase the material strength through grain refinement and precipitation of fine carbo-nitrides, achieved through microalloying and effective heat treatment. Nano-sized carbo-nitrides of microalloying elements can refine austenite grain size during heat treatment as well as strengthen the matrix, where the strength depends on the size, shape, stability and uniformity of the nanoparticles [15]. Si of up to 2% was found to improve spring steel properties through the refinement of tempered carbides [9]. The addition of Nb and V has also been reported beneficial due to the precipitation and dispersion of fine micro-alloyed carbo-nitrides [12,13]. Further improvement can be obtained through the formation of the austenite grain

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substructure [11] as well as through inclusion-assisted nucleation of intragranular ferrite, achieved by introducing fine inclusions, such as titanium oxides [16].

Currently, there is a growing awareness about the potential benefits of nanotechnologies in the modern engineering industry, and a number of investigations are carried out in the area of nanostructured steels [17]. The focus is on the manipulation of microstructure at the nano-scale through innovative processing techniques and adoption of novel alloying strategies. The most promising materials to achieve superior strength and ductility are metal matrix nanocomposites [18]. Superior mechanical properties are attributed to the homogeneous distribution of nano-sized reinforcement particles in the matrix. With nanoparticles reinforcement also high-temperature performance can be improved because of the dispersion of thermally stable ceramic nano-particles [19,20]. At present, several methods are used to produce nano-particles reinforced materials, including the powder metallurgy process based mechanical alloying with high energy milling, a non-conventional vortex liquid metal casting process and ultrasonic cavitation-based solidification [21–23].

However, the main challenge in realizing the immense potential of nano-engineered steels is to manufacture large components and quantities at low costs. Homogeneous dispersion of ceramic nano-particles in molten metals is extremely hard to achieve [24]. The nano-particles tend to agglomerate into coarse clusters during liquid metallurgy processing. This is due to poor wettability of nano-particles with large surface area, which however can be improved by using appropriate synthesis methods. Hydrothermal method has been used for synthesis of hydrophobic nano-particles, i.e. ZnO [25,26]. Homogeneous dispersion becomes more difficult when the specific gravity of molten metal increases, making it difficult to apply for steel. Thus, liquid metal casting processes are mainly restricted to light-weight metal-based composites such as magnesium and aluminum [24]. Consequently, only limited studies exist [24,27,28] which report successful incorporation of nano-sized ceramic particles into the steel using conventional casting routes.

The aim of the present work was to investigate the potential of different nano-particles as alloying elements in the conventional liquid metal casting process and to define their effect on the tensile properties, fracture toughness and fatigue resistance of the spring steel.

## 2. Experimental

#### 2.1. Materials and heat treatment

Commercial 51CrV4 spring steel was used in this investigation as a reference and starting base material, which had the following composition (wt%): 0.51% C, 0.24% Si, 0.95% Mn, 1.07% Cr, and 0.14% V. The steel melt was produced under normal atmospheric conditions by a 20 kg-capacity medium frequency induction furnace. After melting the starting material (19 kg) and adding required ferroalloys to cover melting losses, 10 g of reinforcing nano-particles powder was externally introduced into the melt. Nano-particles included in the investigation comprised ZrC,  $Al_2O_3$  and C-nanotubes powder, all added in the same concentration of 0.05%. Details of nano-powders used are given in Table 1. After the ex-situ introduction of nano-particles (sintered cylinder), the melt was immediately poured into a metallic mold with dimensions of  $60x60 \times 400 \text{ mm}^3$ . Subsequently, the cast ingots were hot rolled to a thickness of 20 mm through 5 passes at 1150 °C and then air cooled.

From hot rolled and soft annealed stripes tensile,  $K_{Ic}$  and CVN specimens were taken in the rolling direction. All specimens were then vacuum heat treated in a vacuum furnace with uniform

high-pressure gas-quenching using nitrogen gas at a pressure of 5 bar. After heating to the austenitizing temperature of 870 °C (10 °C/min), specimens were soaked for 10 min, gas quenched to a temperature of 60 °C ( $\lambda_{800-500}$ =0.42), and then single tempered for 1 h at 475 °C.

#### 2.2. Tensile properties and fracture toughness

Effect of different nano-particles on the mechanical properties of spring steel included tensile test, and measurement of hardness and fracture toughness. Tensile tests at room temperature ( $21 \pm 0.5$  °C; ISO 6892-1) were performed on standard cylindrical specimens with a diameter of 10 mm and gage length of 50 mm using Instron 8802 tensile-test machine. Measurements involved yield strength  $R_{p0.2}$ , ultimate tensile strength  $R_m$ , elongation  $A_5$  and contraction Z.

For fracture toughness measurement circumferentially notched and pre-cracked tension bar specimens (Fig. 1), designated  $K_{Ic}$ -test specimens were used, with the fatigue pre-crack of about 0.5 mm created under rotating-bending loading (600 N, 4500 cycles) before the final heat treatment [29]. Using Instron 8802 tensiletest machine and cross-head speed of 1.0 mm/min load at fracture was measured and fracture toughness  $K_{Ic}$  calculated using Eq. (1) [30]:

$$K_{Ic} = \frac{P}{D^{3/2}} \left( -1.27 + 1.72 \cdot \frac{D}{d} \right)$$
(1)

where *P* is the load at failure, *D* is the outside non-notched diameter (10 mm), and *d* is the diameter of the instantly fractured area. Eq. (1) is valid for 0.5 < d/D < 0.8 and linearly elastic behavior, as displayed by all investigated specimens.

On each cylindrical specimen also the Rockwell-C hardness (HRc) was measured using a Wilson 4JR hardness machine and average values calculated. Up to six measurements were performed on each specimen.

#### 2.3. Fatigue properties

Fatigue properties of reference 51CrV4 spring steel and spring steel alloyed with nano-particles were determined under bending loading. Bending fatigue testing was performed at room temperature on a Rumul Cracktronic resonant machine using Charpy

Table 1Reinforcement nano-particles.

Nano-powder	Size (nm)
ZrC Al <sub>2</sub> O <sub>3</sub> C-nanotubes	60 < 125 10–20
$15$ 10 $L_t=120$	
detail	•

Fig. 1. Circumferentially notched and precracked tension bar specimen.

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