

## SOME NANOSTRUCTURAL ASPECTS OF A MULTIDIRECTIONAL FORGED MICROALLOYED STEEL FOR AUTOMOTIVE AND THEIR EFFECTS

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*The vanadium carbide precipitates in microalloyed steel for automotive crankshaft are investigated using High Resolution Transmission Electron Microscopy (HRTEM). The presence of cubic ordered  $V_8C_7$  nanocarbide has been unequivocally highlighted, after the forging of 38MnVSi6 vanadium microalloyed steel by a new multidirectional flash reduced technology. The literature provides few data relative to the unambiguous presence of  $V_8C_7$  non-stoichiometric carbide, obtained by different other methods in Fe-C-V alloying system. Nanometric carbide precipitates (<3nm) lead to the increase of this HSLA (High-Strength Low-Alloy) steel toughness.*

**Keywords:** vanadium nanocarbide, microalloyed steel, multidirectional flash reduced forging, toughness

### 1. Introduction

About 70% of the strength of the microalloyed steels is given by the type, size and distribution of strengthening precipitates and also by the ferrite-pearlite morphology of the matrix. The strengthening precipitates (e.g. carbides, carbonitrides) are achieved by the strong carbide-forming elements, such as V, Nb and Ti.

A proportion of 0.05 to 0.25% vanadium is the most common microalloying addition in forging steels. The research in the vanadium

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microalloyed steels has been mainly concentrated on stoichiometric cubic vanadium carbide VC [1]. Compared with TiC and NbC, the VC shows an increased solubility in the iron matrix, which allows the dissolution during heating and the fine precipitation during cooling. The vanadium additions produce significant changes in microstructure, such as fraction and lamellar spacing in ferrite as well as precipitation strengthening. The most pronounced effect on strength and elongation properties was found to be obtained by VC precipitation strengthening. The models based on Ashby-Orowan theory, which reveal the effect of VC configuration on bowed-out dislocation behavior, are proposed to describe the strengthening mechanism [2].

Besides the primary vanadium cubic carbide VC, a large series of non-stoichiometric compositions  $VC_x$  with “x” varying from 0.72 to 0.88, has been observed in experiments [3]. This series includes two carbon-vacancy ordered vanadium carbides  $V_8C_7$  and  $V_6C_5$ . Following some long duration heat treatment in Fe-C-V system, the monoclinic ordered  $V_6C_5$  carbide was unambiguously identified by HRTEM investigation. Although many authors have reported the presence of ordered  $V_4C_3$  structure, the diffraction pattern made by Backer and Nutting [4] for this vanadium precipitated carbide corresponds well with the monoclinic ordered  $V_6C_5$  structure [5].

Few data in the literature related to  $V_8C_7$  crystalline structure and to the induced mechanical performance were reported. These data were obtained by various other methods than multidirectional forging of microalloyed steels: the study of the impurity distribution in High Strength Steel Alloys (HSSA), as well as its role in the carbide formation energy and electronic structure studied, by the density functional theory (DFT) [3];  $V_8C_7$  ordered superstructure as an undesirable FEG-TEM irradiation effect in a model Fe-C-V steel, after a long specific two-step heat treatment (10 hours at 700°C and 10 days at 800°C) [5]; in Transformation Induced Plasticity (TRIP) steels, after intercritical annealing and quenching after being held at bainitic isothermal transformation temperature [6].

According to previous work, the size of vanadium carbide precipitates in Fe-C-V system is located in the ultrafine and nano-dimensions (from 10 to 200 nm). In very few cases, the carbides less than 10 nm are successfully obtained, but very long thermal treatments were required [7].

In the field of vanadium microalloyed steels, there are still many controversial and poorly understood subjects that remain open for further investigation [8]:

- the composition and the structure of the vanadium precipitates;
- the interactions between the transition metal carbides with the ferrous matrix (e.g. the homogeneous coherent nucleation of precipitates; the nucleation mechanism for interphase precipitation);
- the mechanism of strain induced precipitation in austenite;

- the influence of both interphase precipitation and random precipitation in ferrite on mechanical properties (eg. yield strength);
- the influence of the processing route parameters on steel properties.

This paper proves the intragranular precipitation of the cubic  $V_8C_7$  carbide 3 nm sized in 38MnVSi6 microalloyed multidirectional flash reduced forging steel. This process has a positive influence on the toughness properties of automotive crankshaft.

## 2. Materials and Methods

Table 1 shows several identification data and the chemical composition of the 38MnVS6 experimental microalloyed steel.

Table 1

Experimental material									
Grade :			38MnVS6						
Number:			1.1303						
Standard:			EN 10267 : 1998 Ferritic-pearlitic steels for precipitation hardening from hot-working temperatures						
Classification:			Non-alloy special steel (standardized); other: Microalloyed steel; HSLA (High-Strength Low-Alloy); AFP (Ausscheidungshärtender Ferritisch-Perlitischer stahl = Precipitation hardening Ferritic-Pearlitic)						
Chemical composition (wt.%)									
Element	C	Si	Mn	P	S	Cr	Mo	V	N
standardized	0.34-0.41	0.15-0.80	1.2 - 1.6	max 0.025	0.02 - 0.06	max 0.30	max 0.08	0.08 - 0.20	0.01-0.02
experimental	0.38	0.54	1.43	0.012	0.03	0.09	-	0.12	-

The material is used for the production of automotive crankshafts. The results of the laboratory investigation on the structure and mechanical characteristics have been used for setting the thermomechanical parameters of the forging process.

Prior to forging, the bars were preheated above 1100°C soaking temperature, then hot forged at 1260°C, from 85 mm<sup>2</sup> to 38-42 mm<sup>2</sup> diameter and finally cooled with 8°C per second rate (in cooled air).

Structural and mechanical investigations have been performed on the samples from the as forged bars and from the forged two-cylinder-crankshafts, by both conventional and new multidirectional flash reduced process.

The technological chain for high duty parts (such as the automotive crankshaft based on flash reduced forging) includes two flashless preformings in closed dies, a multidirectional forging and a final conventional forging operations (Fig. 1). An optimal heating strategy adapted to requirements of the new forging technology (fast induction heating in combination with scale minimisation) has

been applied. The reheating process takes place immediately after the two preforming operations and is followed by multidirectional and final forging.

High resolution structural and elemental analysis were carried out on thin section samples from the steel, by using Transmission Electron Microscopy (TEM) and High-Resolution Transmission Electron Microscopy with Energy Dispersive X-Ray analysis (HRTEM-EDX). Measurements were performed using a TECNAI F30 G2 STWIN transmission electron microscope operated at 300 kV, with field emission gun - FEG with a line resolution of 1.2 Å and an Energy Dispersive X-ray Spectrometer (EDAX) with resolution of 133 eV to MnK $\alpha$ . The cross section samples for TEM analysis were prepared using FISCHIONE ion-milling equipment with Ar ions.

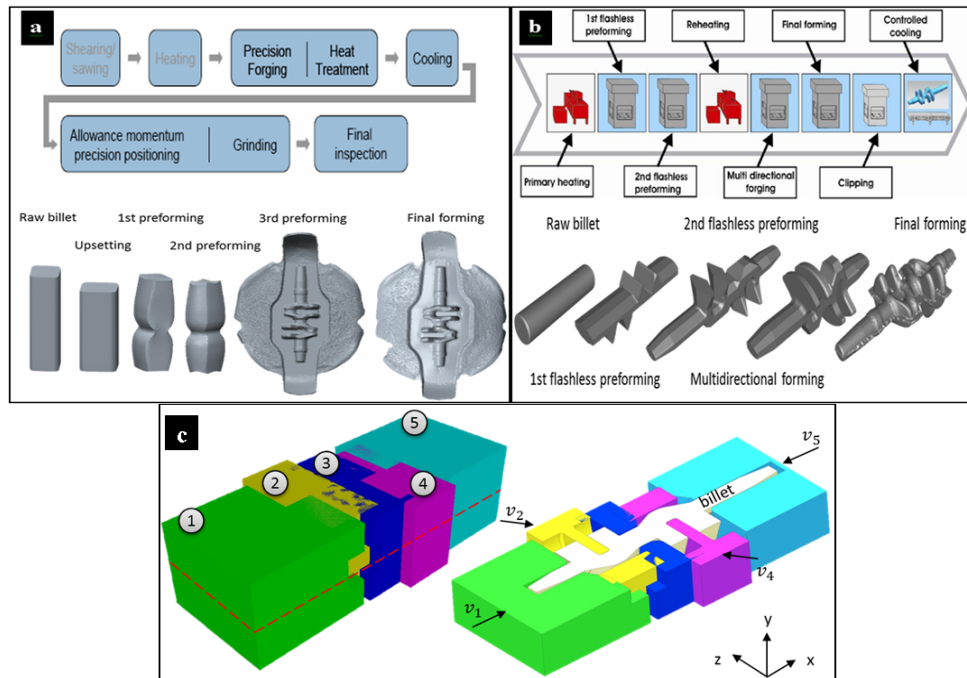


Fig. 1. Conventional (a) and flash reduced (b) process chain for the first two-cylinder-crankshaft. c) Tool design for the multidirectional forming. Left: die numbering. Right: sectional view with moving direction of the dies

The testing of tensile strength was done at 20°C, using longitudinal specimens with 10 mm diameter, according to ASTM E8, ASTM E 21, ISO 6892. The testing equipment was a Universal testing machine EU 40 tf type. Impact bending test was done on 10 x 55 mm<sup>2</sup> cross-section testing bars, according to ASTM E23, ISO 148 and WPM30 type impact testing machine.

Low cycle fatigue test was performed on electro-hydraulic servo-controlled testing machine INSTRON 8801 – 100KN,  $\pm 1\%$  precision of load measurement, high temperature system ( $\pm 20^\circ\text{C}$ ), dynamic extensometer INSTRON HTD,  $L_0=12,5$  mm, class B1 acc. ASTM E 83. The testing parameters for low cycle fatigue have been selected considering: maxim load-5 values, starting with  $0.6\sigma_{\max}$  and decreasing up to  $0.4\sigma_{\max}$ ; asymmetry coefficient  $R = 0.1$  at 5Hz frequency.

The Wöhler curve was determined considering the number of cycles up to the test bar breaking.

Hardness was measured on POLDI automatic tester with 10 mm diameter BrinellBall.

### 3. Results and Discussions

Micro and nanostructural investigations by TEM and HRTEM reveals the presence of pearlite ( $\alpha\text{-Fe} + \text{Fe}_3\text{C}$  plates) major phase (Fig. 2a) and  $\text{V}_8\text{C}_7$  nanoprecipitated carbides.

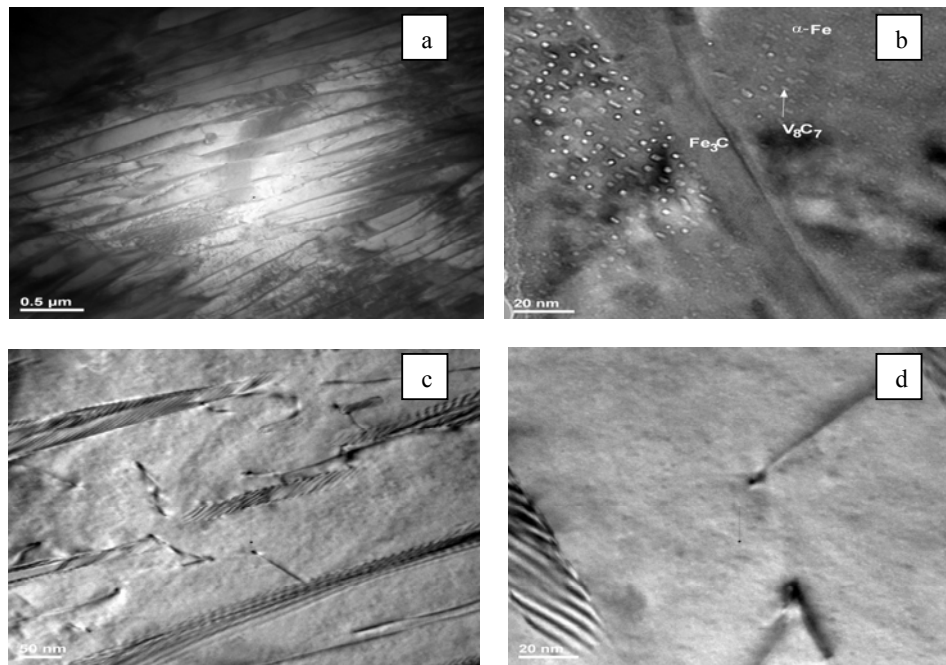


Fig. 2. Bright Field Transmission Electron Microscopy (TEM BF) images of multidirectional forged crankshaft sample: a) pearlitic microstructure ( $\alpha\text{-Fe}$  and  $\text{Fe}_3\text{C}$  plates) and agglomerations of dislocations; b)  $\text{V}_8\text{C}_7$  nanoprecipitates uniformly distributed in pearlitic  $\alpha\text{-Fe}$ ; c) and d) dislocations parallel to the foil surface and pinned in  $\text{V}_8\text{C}_7$  nanoprecipitates

The parameters of deformation and cooling processes during forging (e.g. strain, strain rate and cooling rate, forging and cooling temperature) have favored the presence of 10-20% ferrite and pearlite rests in all investigated cases (i.e. raw as forged material, final forging material by both conventional and flash reduced new technology). Ultrafine ferrite has the submicron grain size and lamellar morphology. The pearlite proportion is controlled by the deformation and cooling parameters, because its growth leads to reduction of the plasticity properties, although the mechanical strength may increase.

The  $V_8C_7$  vanadium nanocarbides are uniformly distributed in pearlitic ferrite ( Fig. 2b), some of them pinning the dislocations (Fig. 2c,d ). The HRTEM images show the presence and distribution of dislocations parallel to the surface of the thin sample (Fig. 2c,d; Fig. 3a) and around the periphery of nanoprecipitates (Fig. 3b). The size of nanocarbides is  $< 3\text{ nm}$  (Fig. 3d).

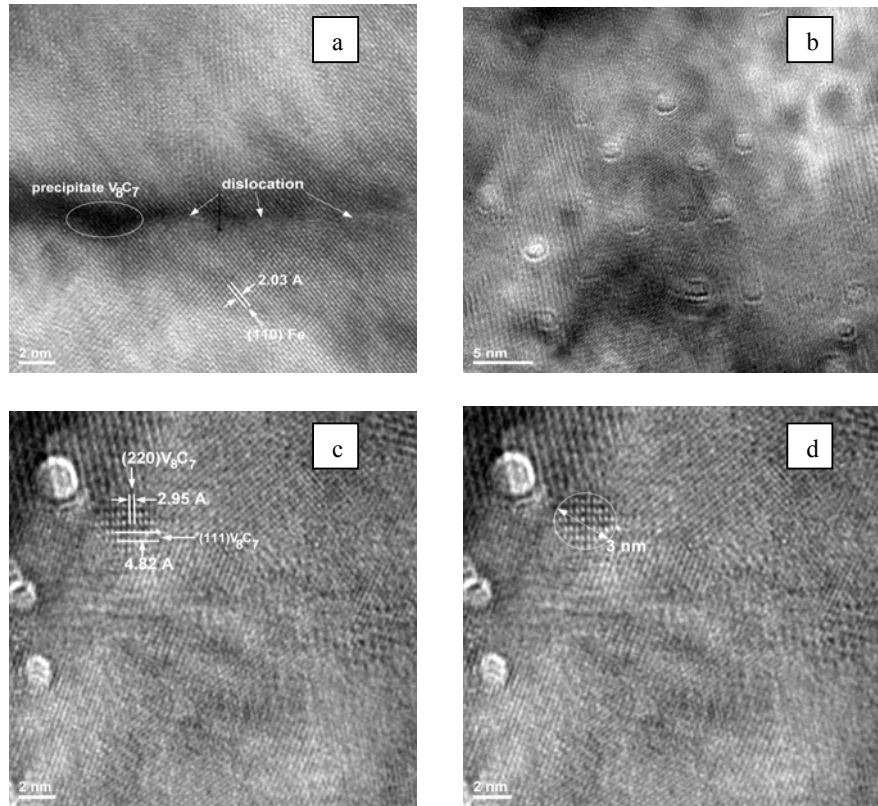


Fig. 3. HRTEM images of multidirectional forged crankshaft sample: **a)** dislocations parallel to the foil surface blocked in  $V_8C_7$  precipitate; **b)** dislocations around the periphery of nanoprecipitates; **c)**  $V_8C_7$  nanoprecipitates in pearlitic  $\alpha\text{-Fe}$ ; **d)** size of  $V_8C_7$  nanoprecipitate.

In Fig.3 a the 2.03 Å interlayer distance of crystalline planes of (110) Miller indexes for  $\alpha$ -Fe is highlighted.

In the Fourier filtering processed HRTEM, the measured interlayer distances of nanocarbide crystalline planes are 2.95 Å and 4.82 Å (Fig. 3c), which are fitting with the distance of planes of (220) and respectively (111) Miller indexes for  $V_8C_7$  cubic crystalline structure (according to 73-0394 ICDD-International Centre for Diffraction Data file).

Energy Dispersive X-ray (EDX) analysis showed that vanadium and carbon microalloying elements are present in nanoprecipitates (Fig. 4).

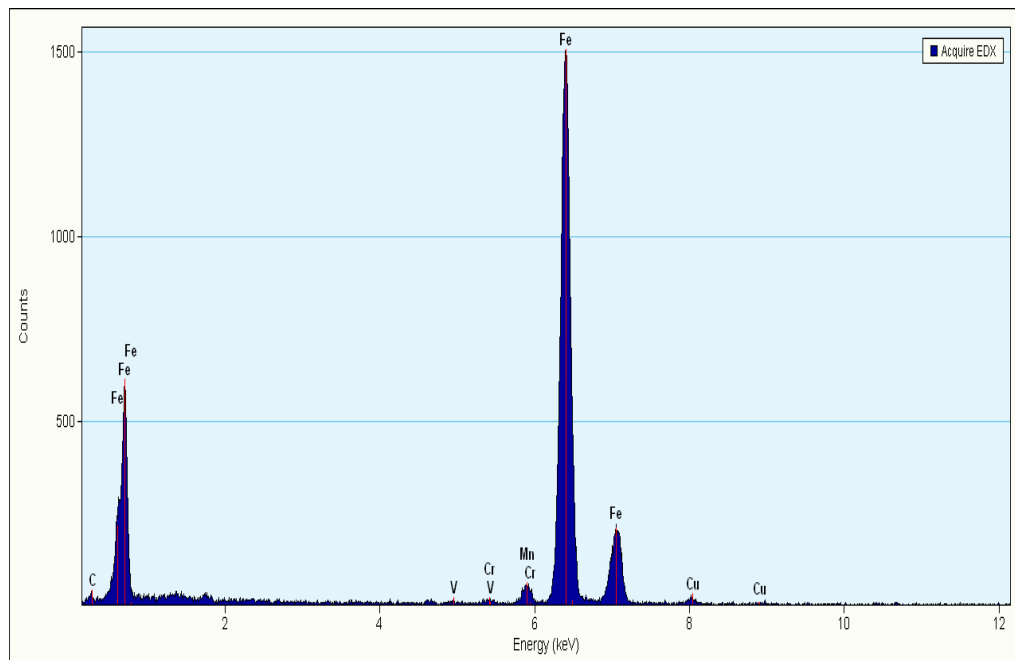


Fig. 4. EDX spectrum recorded in the nanoprecipitates area

Three sample bars have been tested for both strength and impact resistance. The low cycle fatigue test was done on five bars under uniaxial cyclic loading in controlled stress. The results of the tensile test, impact bending test, hardness measurements and low cycle fatigue test are presented in Table 2.

The Wöhler curves for as forged, conventional and multidirectional forged material samples are represented in Fig. 5 and these describe the stress-life correlation, useful in elastic or near elastic range and is addressing situations with constant amplitude loading.

Table 2

Comparative values for 38MnVS6 properties in several stages

Processing state of 38MnVS6 experimental steel	Tensile Strength, (MPa)	0,2 Offset Yield Strength, (MPa)	Elongation A5, (%)	Reduction in cross section on fracture, Z (%)	Impact Energy (J)	Low cycle fatigue		Brinell Hardness	Microstructure
						Load $\sigma_{max}$ (MPa)	No of cycles		
*Bars according EN 10267 in +P condition	800-950	$\geq 520$	$\geq 12$	$\geq 25$	-	-	-	**255	Pearlite + 10 - 30 % ferrite
Bars, in the as forged condition (average values at 20 <sup>0</sup> C)	951	653	7.2	20	26	455	800,000	280	Pearlite + 10 - 20% ferrite
Crankshaft by conventional forging (normalized, average values at 20 <sup>0</sup> C)	990	686	12.33	26.16	30.3	300	1,301,270	295	Pearlite + 10 - 20% ferrite
Crankshaft by new multidirectional flash reduced forging (average values at 20 <sup>0</sup> C)	998	745	10.10	24	21.6	398	4,255,100	320	Pearlite and 10 - 20% ferrite

\*(+P) condition according to DIN EN 10267: Precipitation hardened.

\*\*(+S) condition according to DIN EN 10267: Treated to improve shearability.

The tests results proved that the new multidirectional forged parts have higher strength and ductility properties than the material in as forged and conventional forged conditions.

According to the low cycle fatigue results, the fatigue resistance at  $4 \times 10^6$  cycles and  $R = 0.1$  is:

- in the range of 480-700 MPa for as forged samples;



- in the range of 350-400 MPa for conventional forged samples; for lower  $R = 0$  the fatigue resistance is expected to be lower;
- in the range of 400-500 MPa for the new multidirectional forged samples.

The low cycle fatigue resistance for the new multidirectional technology samples is greater than conventional technology samples.

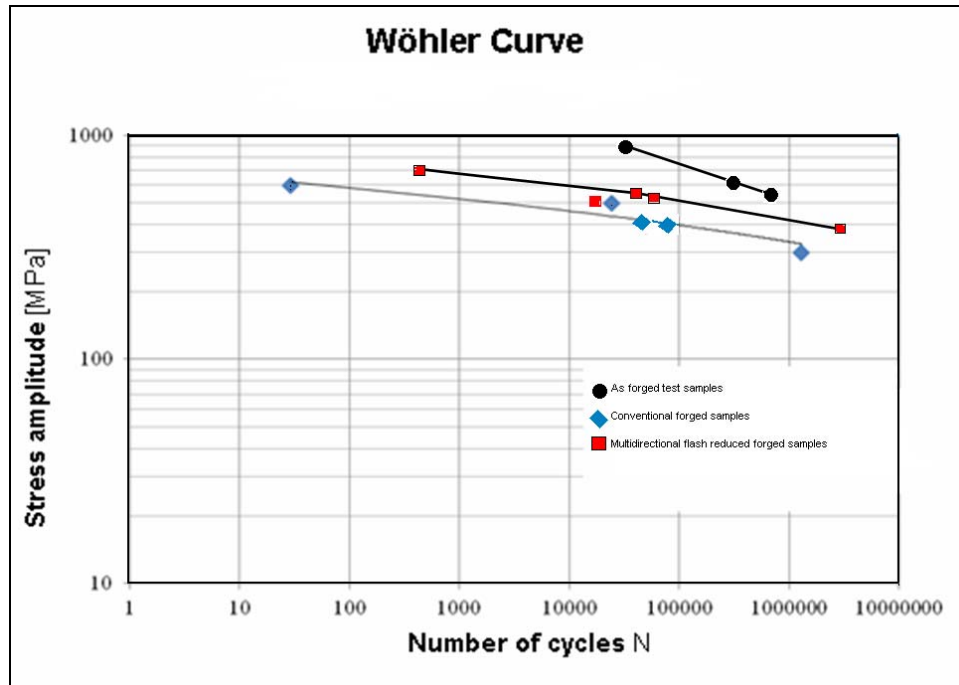


Fig. 5. Wöhler curves for the 38MnVS6 forged in the three experimental stages

The medium carbon microalloyed steel have been developed as a replacement for quenched and tempered low alloy steels, in order to reduce the costs of the wrought components by eliminating the heat treatment sequence (heating, quenching, tempering, stress relieving), whilst preserving acceptable properties and in-service performances.

The 38MnVS6 is a medium carbon microalloyed steel and achieves its properties during the controlled cooling after forging. The hardening mechanism for this steel is different compared to heat treatable steels. As a rule, the vanadium microalloying addition improves the strength by two mechanisms: dispersion strengthening and grain refinement. Mechanical properties are obtained during the

forging/cooling process, while the heating/cooling cycle is critical. The final forging temperature is particularly important, since it affects the austenite grain size and subsequently the tensile strength.

The high strength level in the 38MnVS6 engineering steel can result from a fine nanoparticle dispersion. This can take place by interphase precipitation of vanadium carbides (e.g. interface between austenite and ferrite and between austenite and pearlite). Interphase precipitation of carbides in microalloyed steels has been found to occur in both proeutectoid and pearlitic ferrite [9]. In our case they occur in the pearlitic ferrite. In medium-carbon microalloyed steels the vanadium carbides favor the nucleation of intragranular ferrite [10] and improve the impact and fatigue properties of forged parts by the decrease of the ductile-to-brittle transition temperature.

The multidirectional flash reduced forging of crankshaft leads to increased density of dislocations. The strengthening mechanism can be described by a similar model to that proposed in [2], where the high density of dislocation mainly consists of bowed-out, tangling and looping dislocations. The HRTEM images show that the dislocation structure in ferrite was mainly induced by  $V_8C_7$  precipitates, while the  $V_8C_7$  and cementite in pearlite impede the moving of dislocations during the tensile deformation. As a consequence, the distribution of dislocations in ferrite is more uniform than in pearlite. This phenomenon enhances the strength properties (yield strength, ultimate tensile strength) at levels close to those of Quenched and Tempered (QT) steels, without reducing the ductility properties. In this way, for the AFP precipitation hardening steels the quenching heat treatment can be eliminated and as a consequence the risk of cracks during the treatments is minimized and the financial savings are achieved.

As a general rule, the main routes to achieve the full strengthening potential of microalloying additions in 38MnVS6 microalloyed multidirectional forged steel are as follows:

- preheating prior to forging above 1100°C soaking temperature, to dissolve all vanadium precipitates and then rapid induction heating at 1250°C forging temperature, which allows the dissolution of the microalloying constituents; this process produces the grain refinement of the austenite and increases the ferrite content, which are responsible for a significant increase of ductility and toughness;
- post forging cooling with 8°C per second cooling rate, which produces the transformed ferrite by continuous eutectoid transformation of the austenite during the slow cool from the two-phase  $\alpha$ - $\gamma$  field and which does not inhibit or suppress the precipitation of the strengthening particles such as  $V_8C_7$ ;
- strict adjustment of the chemical composition within/into very restricted ranges, according to Table 2, for the control of the ferrite-pearlite structure, as well as the formation of strengthening precipitates; typical carbon content is

sufficient to form an amount of pearlite, which is responsible for a significant strengthening and also decreases the solubility of the microalloying constituents in austenite; typical vanadium content leads to morphological changes in ferrite microstructure, grain refinement, as well as precipitation strengthening.

#### 4. Conclusions

Using HRTEM, the presence of cubic ordered  $V_8C_7$  nanocarbide (<3nm) has been unequivocally highlighted in 38MnVSi6 microalloyed steel processed by a new multidirectional flash reduced forging.

The tests results proved that the flash reduced forged crankshaft has higher strength and ductility properties, higher impact bending and low cycle fatigue, compared to the conventional process. As a rule, the dislocation structure in transformed ferrite and pearlite induced by  $V_8C_7$  precipitates leads to an increase of the strength properties, whilst the ductility properties are preserved.

In common forging processes for complicated high duty parts, especially crankshafts, an excess on material (flash) is technically needed, which results in a material utilization between 60 and 80 %. The new multidirectional flash reduced forging drops significantly this excess of material, increasing the material utilization up to 90 %.

The induction reheating of complicated geometries within a forging line represents a new approach in the forming processes.

The combination of the new forging sequence and the induction heating procedures has a large technological and economic potential for the production of high quality work pieces, enhancing significantly the state-of-the-art in this area.

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