

# **EFFECT OF VANADIUM AND THERMOMECHANICAL TREATMENT ON THE PROPERTIES OF 55SiCr6**

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## **ABSTRACT**

The microstructure and tensile properties of conventionally heat treated and thermomechanically treated 55SiCr6 with or without vanadium were investigated. By using optimized thermomechanical treatment the ductility was improved and at the same time the tensile strength could be increased up to 37%. To define the conventional heat treatment and thermomechanical treatment parameters the austenitization conditions and the recrystallization behaviour for a logarithmic strain of  $\varphi=0.4$  and a quench delay of 15s were investigated. Conventionally heat treated samples were austenitized at 900°C, quenched and tempered between 250 and 450°C. Single step thermomechanically processed samples were austenitized at 900°C, deformed between 900 and 750°C with  $\varphi = 0.4$ , quenched and tempered between 300 and 400°C. Furthermore, two step thermomechanical treatments with two deformations prior to quenching were carried out with and without vanadium.

The addition of 0.150 mass% vanadium decreases the austenite grain size below 950°C and 15 minutes holding time. Additionally it increases the recrystallization temperature from 800 to 850°C. The vanadium free silicon chromium steel shows a minimum in ductility at tempering temperature of 350°C. No minimum was observed for 55SiCrV6. Without vanadium the improvement of the ductility for constant tempering temperature by thermomechanical treatment is pronounced. A two step thermomechanical treatment of the vanadium free steel results in the highest ductility after tempering at 300°C. The steel microalloyed with vanadium and thermomechanically treated presents the best combination of strength and ductility after tempering at 300 and 350°C.

Scanning electron microscopy shows that spheroidization of carbides start at tempering temperatures of 400°C and above. After tempering at 350°C fine carbide films are observed for conventionally heat treated 55SiCr6.

It is possible to increase the strength and ductility of conventionally heat treated samples by addition of vanadium or by thermomechanical treatment. The highest improvement is achieved though by combining both thermomechanical treatment and microalloying.

## **KEYWORDS**

Thermomechanical treatment, austenite conditioning, microalloying, vanadium, austenite grain refinement, recrystallization, carbide precipitation.

## INTRODUCTION

In all light-weight structures weight reduction is the main objective. Depending on the design profile for an individual part the materials have to fulfill specific design requirements that are in most cases contradictory to each other. Moreover, the cost for a certain weight reduction must be under a limit that is defined by the application itself. In the aerospace sector for example, the prices paid for weight reduction are higher than in the automotive sector.

Steels for automotive springs are such examples where higher strength is demanded and where the design requirements are contradictory to each other. The demand for a stronger material is combined with high ductility, toughness, fatigue, and corrosion requirements. Silicon chromium steels with carbon contents between 0.55 and 0.6 mass % carbon, that have been introduced in the 90's, exhibit a high strength and high sag resistance. Today, the ultimate tensile strength of those silicon chromium steels used for coil springs is about 2000 MPa that corresponds to a tempering temperature around 380°C. An optimized processing, control of the impurity elements and control of the austenite grain size prior to quenching leads to a reduction of area of approximately 40%. A strength improvement by reduction of tempering temperature would reduce the ductility to unacceptable levels [1]. Furthermore, the reduction of ductility is intensified by presence of impurity elements such as phosphorous or copper and tin.

The optimization of strength-ductility combination can either be achieved by variation of the chemical composition or by introduction of a new process. In this paper we firstly investigate the effect of vanadium on the tensile properties of conventionally heat treated silicon chromium steels and secondly present results for thermomechanically treated silicon chromium steels with and without vanadium. The change of mechanical properties is explained by the change of microstructure.

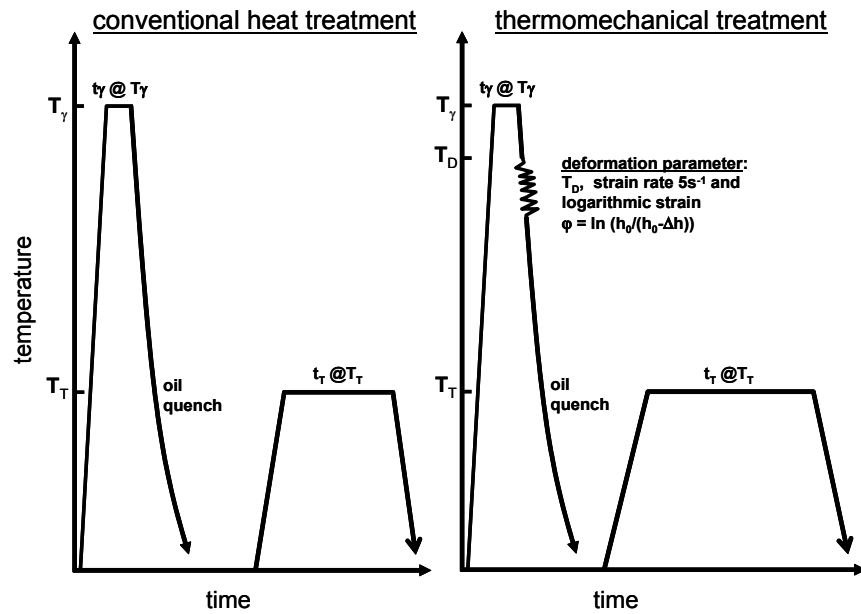
## EXPERIMENTAL

Two ingot of 70kg weight each and a cross section of 140x140mm<sup>2</sup> were obtained by vacuum induction melting. The composition of both melts is presented in table 1. Bars were cut out of the ingot with a cross section of 70x70mm<sup>2</sup>. They were twice homogenized at 1100°C for one hour and then air cooled. After each homogenization operation the bars were hot-rolled. Bars with final thickness of 21 and 15mm thickness and width of approximately 60mm were produced. Cuboidal samples for conventional (cross section RD plane  $A_{\text{CHT}}=55 \times 12.4 \text{ mm}^2$ ) and thermomechanical treatment ( $A_{\text{TMT}}=55 \times 18.4 \text{ mm}^2$ ) were machined from the bars.

The conventional heat treatment and the thermomechanical treatment were conducted using the large-scale 2.5MN hot press at the Max-Planck-Institut für Eisenforschung. These treatments are presented schematically in Fig 1 and the parameters are given in table 2. Heating to austenitization temperature was carried out by induction heating. For all thermomechanical treatments the rolling was simulated by the plane strain compression test in the hot press with a strain rate of 5s<sup>-1</sup>. The tempering was carried out in a furnace.

**Table 1** Chemical composition of investigated silicon chromium steels (mass %)

Melt	C	Si	Mn	P	S	Cr	Cu	Sn	V	Fe
55SiCr6	0.55	1.56	0.59	0.0023	0.023	0.69	0.281	0.030	-	bal.
55SiCrV6	0.56	1.47	0.66	0.0025	0.022	0.69	0.285	0.031	0.163	bal.



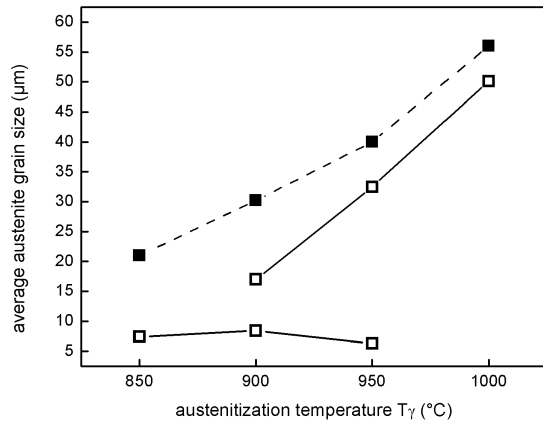
**Fig. 1** Schematic presentation of a treatments used;  $T_\gamma$  austenitization temperature,  $T_D$  deformation temperature,  $T_\tau$  tempering temperature

The austenitization conditions were investigated using a dilatometer. Cylindrical samples with 5 mm diameter and 10 mm height were employed. The longitudinal direction of the samples was parallel to the normal direction of the bars. Two different approaches were applied to determine the prior austenite grain size. The first was standard heat treatment of the cylindrical samples consisting of heating to austenitization temperature, quenching and optical microscopy of samples. The second was using the thermal etching method to reveal the prior austenite grains. The cylindrical samples were grinded and polished in a sample holder along their longitudinal direction. The polished samples were then heated to austenitization temperature and held at this temperature for 15 minutes. During quenching the samples were exposed to a gas mixture containing oxygen. This treatment leads to etching of the grain boundaries and prior austenite grain boundaries can immediately be observed in an optical microscope without chemical etching. Furthermore, the surface relief formed during martensite transformation can be observed (Fig. 3 to Fig. 5).

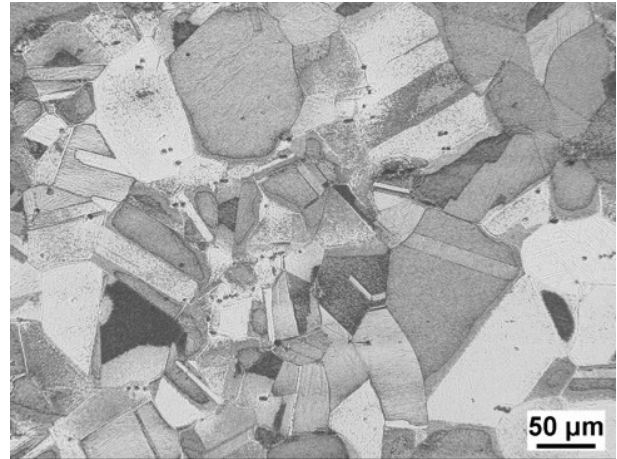
After termination of heat treatment or thermomechanical treatment round tensile specimen were machined from the samples in the rolling direction. Tensile tests were performed at room temperature. Sample for microstructure investigations were cut and prepared using standard grinding and polishing techniques. Etchants used for optical microscopy and scanning electron microscopy were nital (1% alcoholic  $\text{HNO}_3$  solution) and saturated picral solution.

**Table 2** Parameter for conventional heat treatment (no deformation) and for thermomechanical treatment

Melt	Austenitization parameter		deformation parameter		Tempering parameter	
	time $t_\gamma$ (min)	temperature $T_\gamma$ (°C)	strain $\epsilon$ (-)	temperature $T_D$ (°C)	time $t_\tau$ (min)	temperature $T_\tau$ (°C)
55SiCr6	5	900	-	-	60	250, 300, 350, 400, 450
			0.4	850		300, 350, 400
			0.4	750		
			0.4 + 0.4	850 + 750		300
55SiCrV6	5	900	-	-	60	250, 300, 350, 400, 450
	5	920	0.4 + 0.4	900 + 850	60	300, 350



**Fig. 2** Average prior austenite grain size of 55SiCr6 and 55SiCrV6 after 15 minutes austenitization at austenitization temperature  $T_\gamma$

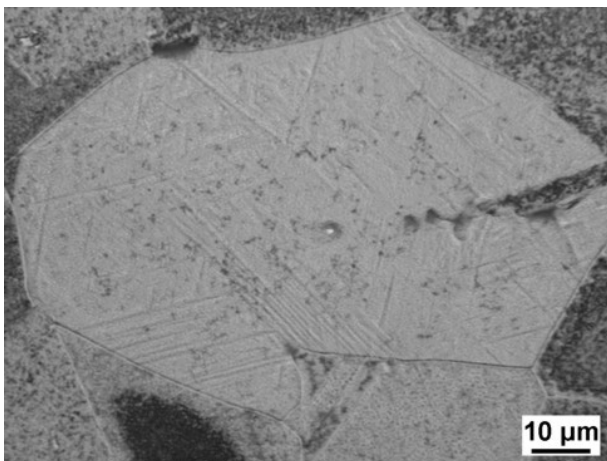


**Fig. 3** Prior austenite grains of thermally etched sample of 55SiCrV6 after holding 15 minutes at 1000°C and quenching

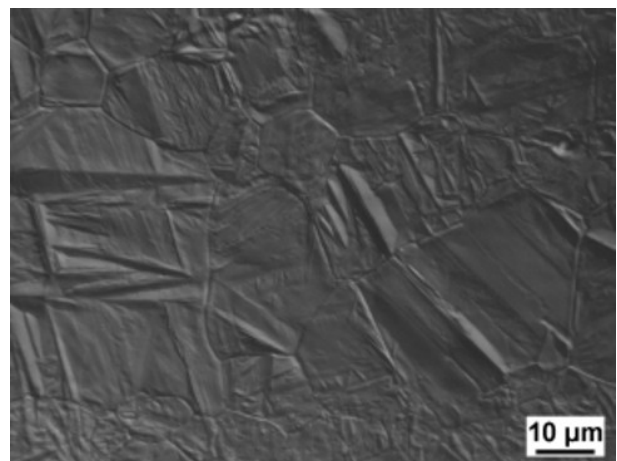
## RESULTS & DISCUSSION

### GRAIN SIZE

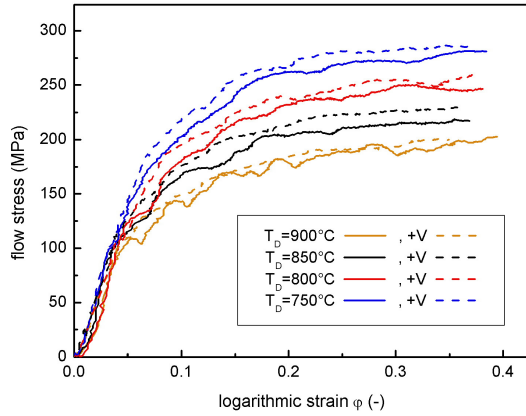
The average prior austenite grain size for different austenitization temperatures and 15 minutes austenitization time is plotted in Fig. 2. Addition of 0.163 mass % vanadium reduces the grain size in the austenitization range between 850 and 1000°C. Between 900 and 950°C there is a bimodal grain size distribution (Fig. 5) and this indicates that not all the vanadium carbides are dissolved during austenitization below 950°C. According to the formula proposed by Turkdogan [2] for a carbon concentration of 0.55% vanadium carbides are thermodynamically not stable above 930°C. At 950°C the bimodal distribution disappears after holding 30 minutes at austenitization temperature and only large grains were observed. The dissolution of vanadium carbides is controlled by kinetics and hence sufficient time for diffusion is necessary. The bimodal grain size distribution is observed, because only a fraction of grains is hindered by the remaining vanadium carbides. Even at 1000°C the time needed for dissolution of carbides expresses itself in the smaller average grain size. Before the dissolution of the carbides, the grains are pinned by the undissolved carbides and growth is slowed down. Therefore a smaller average grain size is even observed after austenitization at 1000°C. At 850°C enough vanadium carbides are present and only small grains are observed.



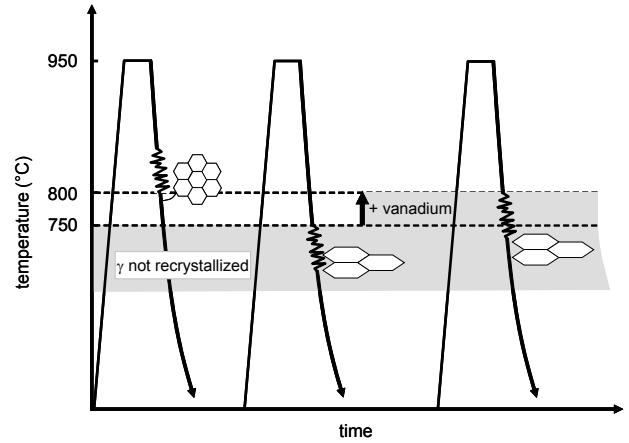
**Fig. 4** Prior austenite grains of thermally etched sample of 55SiCrV6 after holding 15 minutes at 1000°C and quenching



**Fig. 5** Prior austenite grains of thermally etched sample of 55SiCrV6 after holding 15 minutes at 950°C and quenching



**Fig. 6** Flow curves for 55SiCr6 and 55SiCrV6 at deformation temperatures between 750 and 900°C. Measured in plane-strain compression.

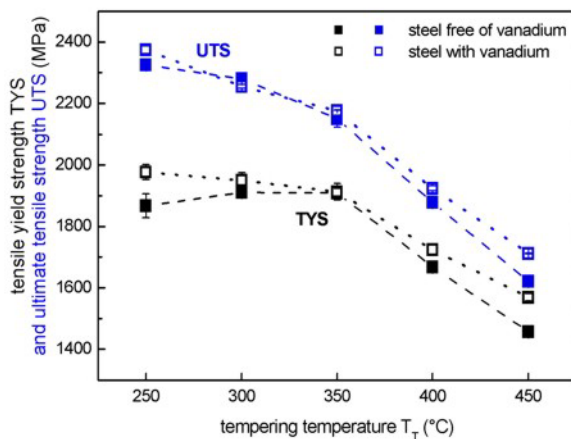


**Fig. 7** Austenite grain structure after deformation with a logarithmic strain of  $\phi=0.4$ , strain rate  $5s^{-1}$  and 15s between deformation and quenching

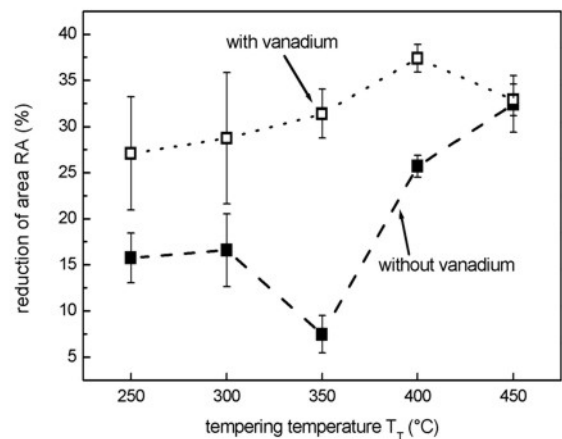
## RECRYSTALLIZATION

Samples from both melts were austenitized for 5 minutes at 950°C and then deformed at various temperatures prior to quenching. The deformation strain of 0.4 and strain rate of  $5s^{-1}$  were kept constant for all tests. The samples were quenched 15 s after deformation was initiated. In Fig. 6 the flow curves for samples with and without vanadium are plotted for all the deformation temperatures tested. The microalloyed steel with vanadium exhibits slightly higher flow stresses at deformation temperatures below 900°C. Optical microscopy was performed after quenching and tempering to evaluate the austenite state before quenching.

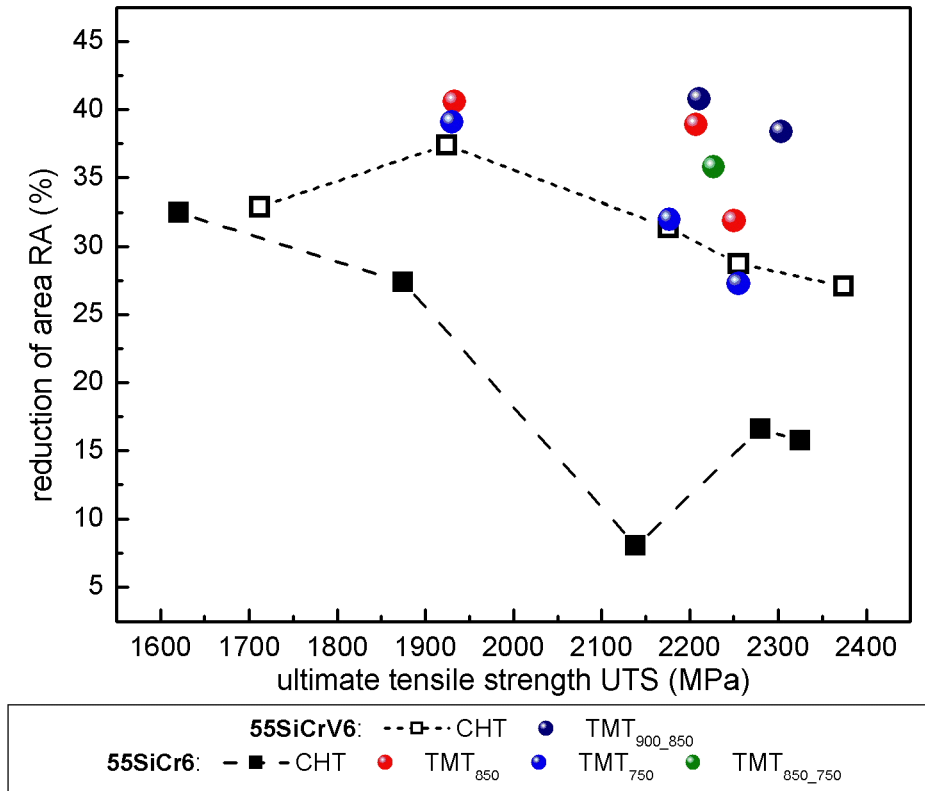
Fig. 7 summarizes the results in schematic manner. Because of the short austenitization time at 950°C not all the vanadium carbides are dissolved. Thus after deformation the deformed austenite grains are stabilized and recrystallization is retarded. Hence, for the same deformation conditions an increase of recrystallization temperature was observed for the vanadium microalloyed steel. The vanadium addition increases the temperature range within which the austenite is not recrystallized prior to quenching.



**Fig. 8** Tensile yield strength TYS and ultimate tensile strength UTS for different tempering temperatures  $T_T$  and for 55SiCr6 and 55SiCrV6.



**Fig. 9** Reduction of area for different tempering temperatures  $T_T$  and for 55SiCr6 and 55SiCrV6.



**Fig. 10** Strength-ductility combination of conventionally heat treated and thermomechanically treated (TMT) samples of 55SiCr6 and 55SiCrV6.

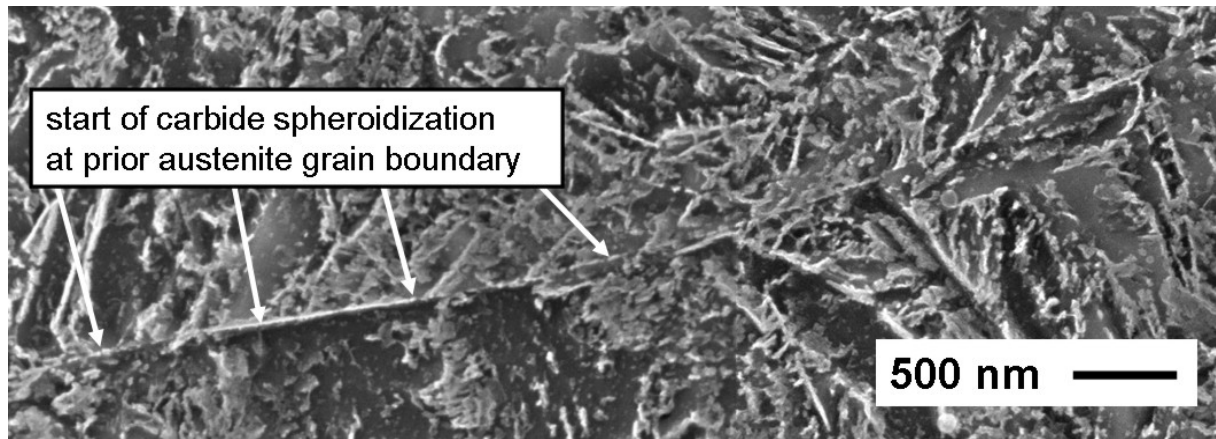
## MECHANICAL PROPERTIES

Mechanical properties after conventional heat treatment for both steels are presented in Fig. 8 and 9. The microalloyed steel exhibits higher tensile yield strength and ultimate tensile strength at the upper range of the investigated tempering temperatures. The difference in strength values at a tempering temperature of 450°C is approximately 100 MPa.

Microalloying with vanadium leads to significantly higher reduction of area values for conventional heat treatment. The vanadium free steel has a minimum value of ductility at 350°C (Fig. 9). The microalloyed steel shows no minimum, the ductility decreases continuously from 400 to 250°C. The higher ductility is a result of the smaller average austenite grain size that was generated at the austenitization temperature of 900°C (Fig. 2).

To investigate which austenite modification results in an improved strength-ductility combination two austenite modifications corresponding to two different deformation temperatures were selected for the vanadium free steel. The selection of the deformation temperatures was based on the recrystallization investigations described above (Fig. 7) and results presented in [3]. Deformation temperature of  $T_D=850^\circ\text{C}$  was selected for a recrystallized austenite modification, and  $T_D=750^\circ\text{C}$  for the non-recrystallized, pan-caked modification. A sequence of two deformations, first deformation at 850°C and second at 750°C was defined for optimum mechanical properties (see table 1). For the vanadium microalloyed steel a combination of two deformations in the recrystallization range was defined for thermomechanical treatment.

Fig. 10 summarizes the results of the tension tests for various treatments. The presence of vanadium increases the ductility for the conventional practice that consists of quenching from the austenite

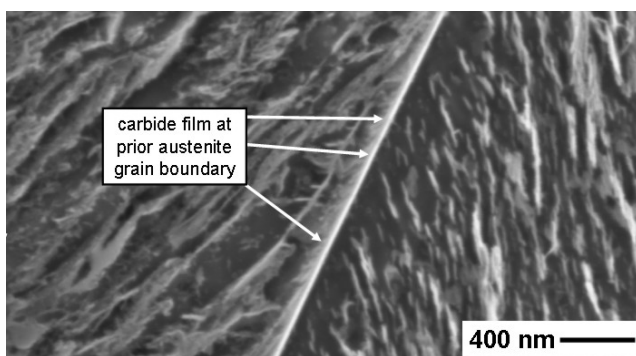


**Fig. 11** Conventionally heat treated 55SiCr6, tempered at 400°C. Etched with nital (1% alcoholic HNO<sub>3</sub> solution)

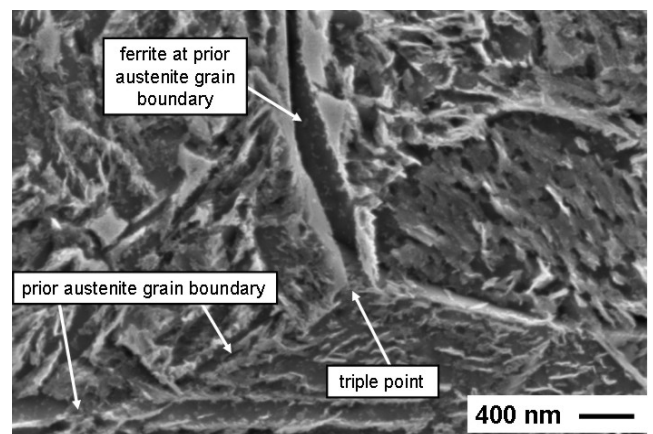
region and tempering. However, the same level of ductility improvement can be achieved by thermomechanical treatment (Fig. 10). Both austenite modifications, the recrystallized and the non-recrystallized one increase the ductility of the silicon chromium steel significantly. For the same tempering temperatures the ductility level is equal or above that of the conventionally heat treated steel microalloyed with vanadium. The results show that an improvement of strength-ductility combination for 55SiCr6 is possible by microalloying with vanadium or by thermomechanical treatment. Taking the reduction of area of conventionally heat treated samples of 55SiCr6 that are tempered at 450°C as reference, then the strength can be increased above 600 MPa while maintaining the same level of ductility or even improving it. Further increase of the strength-ductility combination is possible, if the microalloyed steel is subjected to a two step thermomechanical treatment with deformations at 900°C and 850°C (TMT<sub>900\_850</sub>, Fig. 10).

#### CARBIDE PRECIPITATION AT PRIOR AUSTENITE GRAIN BOUNDARIES

It was shown in [3] that for a 55SiCr6 with 0.54 mass % copper and 0.05 mass % tin the minimum in ductility for conventionally heat treated samples at 350°C is concurrent with the presence of carbide films at the prior austenite grain boundaries. The carbide morphology and distribution at prior austenite grain boundaries of conventionally heat treated samples (with and without vanadium) was investigated for different tempering temperatures using scanning electron microscopy. At tempering temperatures of 400°C and above the carbides start to spheroidize (Fig. 11). Carbide films are observed at prior austenite grain boundaries for the



**Fig. 12** Conventionally heat treated 55SiCr6, tempered at 350°C. Etched with nital (1% alcoholic HNO<sub>3</sub> solution).



**Fig. 13** Conventionally heat treated 55SiCrV6, tempered at 350°C. Etched with nital (1% alcoholic HNO<sub>3</sub> solution).

conventionally heat treated vanadium free silicon chromium steel that were tempered at 350°C (Fig. 12). After quenching and tempering at 350°C the vanadium microalloyed steel is not only decorated with carbide films at grain boundaries but with ferrite islands as well (Fig. 13). The presence of ferrite islands at the grain boundaries might be a result of the undissolved vanadium carbides that locally reduce the concentration of the carbon in the austenite and hence increase the  $A_{r3}$  temperature.

## CONCLUSION

In this paper two ways of optimizing the strength and ductility combination of a silicon chromium steel were presented. Microalloying with vanadium and use of appropriate austenitization conditions leads to a refinement of the austenite grain size prior to quenching. The refined austenite grain size results in an improved ductility for tempering temperatures between 250 and 450°C. The improved combination of strength and ductility can be obtained as well by various thermomechanical treatments. It was shown, that a recrystallized and a non-recrystallized austenite modification, both improve the ductility. The combination of microalloying together with thermomechanical treatment gives the best mechanical properties for the composition presented here.

## ACKNOWLEDGEMENT

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