ETCHING TECHNIQUES FOR THE MICROSTRUCTURAL CHARACTERIZATION OF COMPLEX PHASE STEELS BY LIGHT MICROSCOPY

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Abstract

In this work different etching techniques were applied in order to characterize the microstructure of the complex-phase steel grade CP600 by light microscopy. To enhance the contrast between the phases, different etchants were used consecutively and different etching methods were combined. The amount of retained austenite was measured by a magnetic-volumetric method. To confirm the results of the studies, microhardness and standard hardness tests were made.

Key words

advanced high strength steels, complex-phase steels, microstructure, etching, light microscopy, phase identification

INTRODUCTION

Recent developments in the automotive industry focused on the design of high strength steels. The challenge is to reduce car body weight and CO_2 emissions while increasing the passenger safety. This leads to the demand for stronger and more ductile materials like AHSS (Advanced High Strength Steels). In a cooperation between automotive manufacturers, steel industry and research institutes the ULSAB-AVC program (Ultra Light Steel Auto Body - Advanced Vehicle Concept) devised an AHSS auto body which is 25% lighter than of a bench-marked car [1, 2].

The AHSS are multi-phase steels consisting of hard phases like martensite, bainite and retained austenite embedded a soft ferritic matrix.

Besides the mechanical properties like strength and formability, knowledge about the microstructure of these materials is very important, since it could enable a rough estimate about the mechanical properties. Techniques like light (optical) microscopy (LOM) or scanning electron microscopy (SEM) are established for the visualization of the microstructure. The advantage of LOM over SEM is the visualization of surface layer and not just a surface topography. Due to this LOM feature, it is possible to use not only grain boundary etchings, but also surface layer etchings and the combination of the different etching techniques for the visualization of the microstructure.

EXPERIMENTAL PROCEDURE

An industrially produced complex-phase steel (strength level 600 MPa) was investigated. Its chemical composition as well as its mechanical properties are listed in Table **1**.

CHEMICAL COMPOSITION AND MECHANICAL PROPERTIES OF CP600 PRODUCED BY VOESTALPINE STAHL GmbH

Table 1

Grade	Chemical composition [wt.%]						$R_{p0,2}$ [MPa]	R_m [MPa]	A_g [%]	A [%]
	C	Si	Mn	Cr	P	Mo				
CP600	0,10	0,14	1,51	0,80	0,009	0,004	476	635	11	20

The specimens were wire cut from the sheet material, parallel (longitudinal) and transverse to the rolling direction as shown in **Fig. 1**.

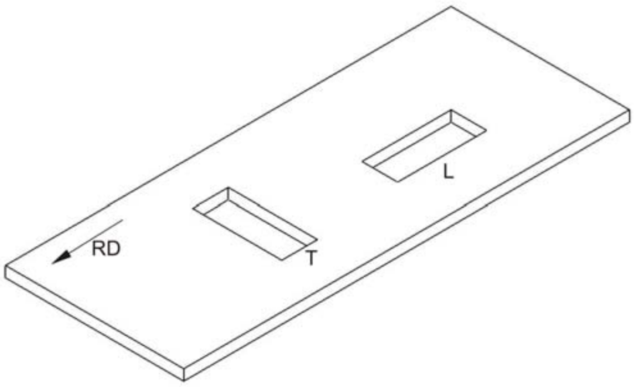

Fig. 1. *Specimens taken longitudinal (L) and transverse (T) to the rolling direction (RD)*

The specimens were embedded in epoxy resin, ground and polished. Polishing was performed semi-automatically with water cooling down to a grit of 1 µm. A defined area was marked with a diamond pyramid mount in a microscope instead of an objective lens. The specimens were etched and the marked area was analyzed. After each analysis the specimen was gently polished and etched with a different etchant again. By using this technique, it is possible to compare the same microstructure using different etchings. The etchant composition and etching principles are listed in Table 2.

Light (optical) microscopy (LOM) was used to analyze the microstructure. The LOM images were taken at a magnification of 1500x. Line intercept method was applied for quantitative description of the microstructure of the investigated steel grade etched with Nital + $Na_2S_2O_5$. The analysis was done with a line distance of 5 µm and an area of 50.000 µm^2 (10 images). This area and a line length of 10.000 µm provide a statistical security [5].

The volume fraction of retained austenite was determined via magnetic-volumetric measurements (Joch-Isthmus method, saturation magnetification) [6].

After micro analysis, microhardness and hardness measurements were conducted. In grains of ferrite and bainite/tempered martensite (TM) microhardness measurements were performed with an indentation load of 19,6133 mN, a penetration speed of 1,96 mN/sec and a holding time of 15 seconds. To obtain a mean hardness of the material and to verify the microhardness measurements a standard hardness measurement (Vickers HV10) was applied. The measurements were conducted according to DIN EN ISO 6507-1.

LISTING OF USED ETCHANTS [4] Table 2

Etchant	Composition	Etching principle
Nital	100 ml ethanol 99%, 10 ml nitric acid 65%	Grain boundary etching
LePera	50 ml $Na_2S_2O_5$ 1% in aqueous dilution, 50 ml picric acid 4% in ethanol	Anodic surface layer etching
Beraha I	1 g potassic sulphite, 100 ml parent dilution Beraha I (1000 ml distilled water, 200 ml hydrochloric acid 32%, 24 g ammonium hydrogen-difluoride)	Anodic surface layer etching
Kalling I	33 ml destilled water, 33 ml ethanol 99%, 33 ml hydrochloric acid 32%, 1,5 g copper(II)-chloride	Cathodic surface layer etching
Nital + $Na_2S_2O_5$	preetching with Nital, wet etching with $Na_2S_2O_5$ 10% in aqueous dilution	Grain boundary and anodic surface layer etching

RESULTS AND DISCUSSION

Fig. 2 shows the steel CP600 differently etched. Images of the microstructure etched with LePera, which forms on ferrite a light brown, on martensite a white, on tempered martensite a brown structured, on bainite a dark structured surface layer and on pearlite a dark surface layer, are shown in Fig. 2a. The application of this etchant enabled to distinguish ferrite from martensite but it did not allow the distinct identification of tempered martensite, bainite and retained austenite.

To identify all existing phases, consecutive etchings were applied. The images of the microstructure etched with Beraha I, Kalling I and Nital + $Na_2S_2O_5$ are shown in Fig. 2b-d. As a reference point the circle marks the same grain in all images. All three etchings color ferrite white and martensite black or dark brown. Beraha I is additionally coloring bainite and tempered martensite light brown. By a combination of Nital and $Na_2S_2O_5$, tempered martensite and bainite both appear brown structured and pearlite becomes black (though in a negligible amount).

After phase identification, a quantitative phase analysis after Nital + $Na_2S_2O_5$ etching was performed. This etching provides a high contrast of the grain boundaries because of the combination of a grain boundary etching with an anodic surface layer etching. The results of the phase analysis are listed in Table 3. The content of retained austenite was determined magnetically.

RESULT OF THE PHASE QUANTIFICATION Table 3

Specimen	Ferrite [%]	Martensite [%]	Retained austenite [%]	Bainite / TM [%]
CP600	54	6,9	0,3	38,8

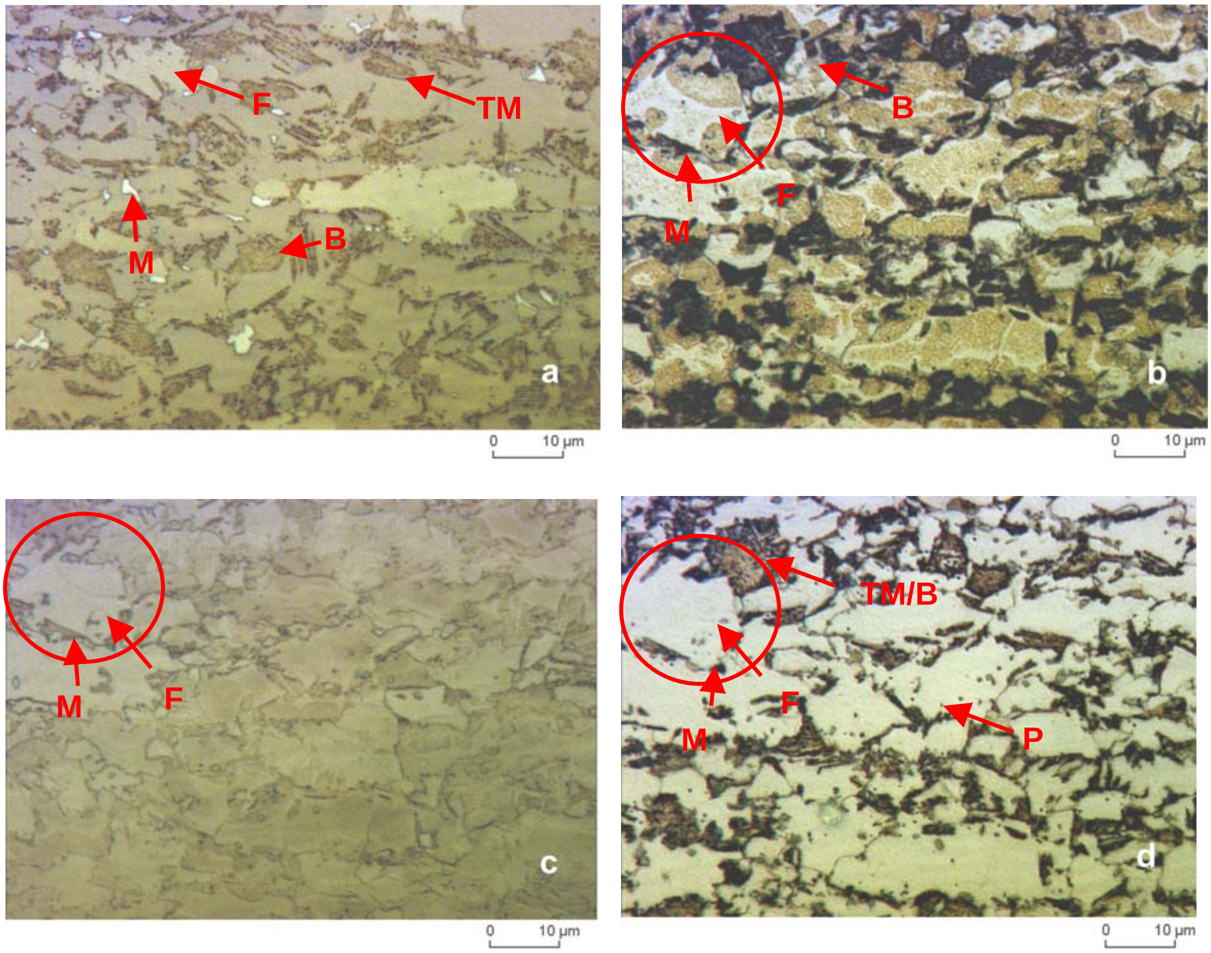

Fig. 2. *Optical micrographs of CP600 etched with (a) LePera, (b) Beraha I, (c) Kalling I, (d) Nital+* $Na_2S_2O_5$*, (B=bainite, F=ferrite, M=martensite, TM= tempered martensite, P=pearlite)*

The microhardness measurements (see Table 4) show that bainite/TM is approximately three times harder than ferrite. Part of the hardness difference between the bainite/TM and the ferrite grains is caused by grain boundary effects. Because of the small grain size, the indentation is too close to the grain boundaries and the measurement detects the hardening effect of the grain boundaries. A measurement with a lower load (ultra-microhardness) would remedy the grain boundary effects. The results also show that the hardness of the ferrite grains and the mean hardness longitudinal and transverse to the rolling direction are nearly the same, see Table 4.

RESULTS OF THE MICROHARDNESS AND THE HARDNESS MEASUREMENTS LONGITUDINAL (CP600 L) AND TRANSVERSE (CP600 T) TO THE RD Table 4

Specimen	Ferrite [HV0,002]	Bainite / TM [HV0,002]	HV10 [HV10]
CP600 L	207,6	549,6	209,7
CP600 T	203,6	596,8	211,3

SUMMARY

An industrially produced complex-phase steel grade with a strength of 600 MPa was investigated. To identify the phases consecutive etchings were applied on a marked specimen area with Beraha I, Kalling I and Nital + $Na_2S_2O_5$. A combination of Nital and $Na_2S_2O_5$ was used to enable a quantitative analysis of the microstructure consistence because it provides a sufficiently high contrast of the grain boundaries. Material with finer microstructure is hard to analyze, because of the maximum possible magnification of 1500x in the LOM. The microhardness measurement gives reliable results only for rather large ferrite grains. Therefore SEM analysis combined with electron diffraction (EBSD) and ultra-microhardness measurements should be considered.

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