

## Identification of Virgin Martensite in Quenched Microstructures of New Generation of Forging Steels with Higher Silicon Content Using Colour Etching

Štěpán Jeníček, Ivan Vorel, Michal Pekovič

Regional Technological Institute, University of West Bohemia in Pilsen, Univerzitní 22, 306 14 Plzeň. Czech Republic. E-mail: jeniceks@rti.zcu.cz, frost@rti.zcu.cz, pekovicm@rti.zcu.cz

Observation and identification of products of transformation of austenite during austempering, Q-P processing, or quenching and tempering are often challenging. The reason is that the resulting microstructures are typically very fine and provide insufficient contrast between microstructural components. Their analysis requires scanning or transmission electron microscopy which demand rather complex sample preparation procedures and involve high costs of maintenance of microscopes and accessories. However, in-process inspection of products of heat treatment calls for simpler and rapid methods of microstructure analysis using a light microscope.

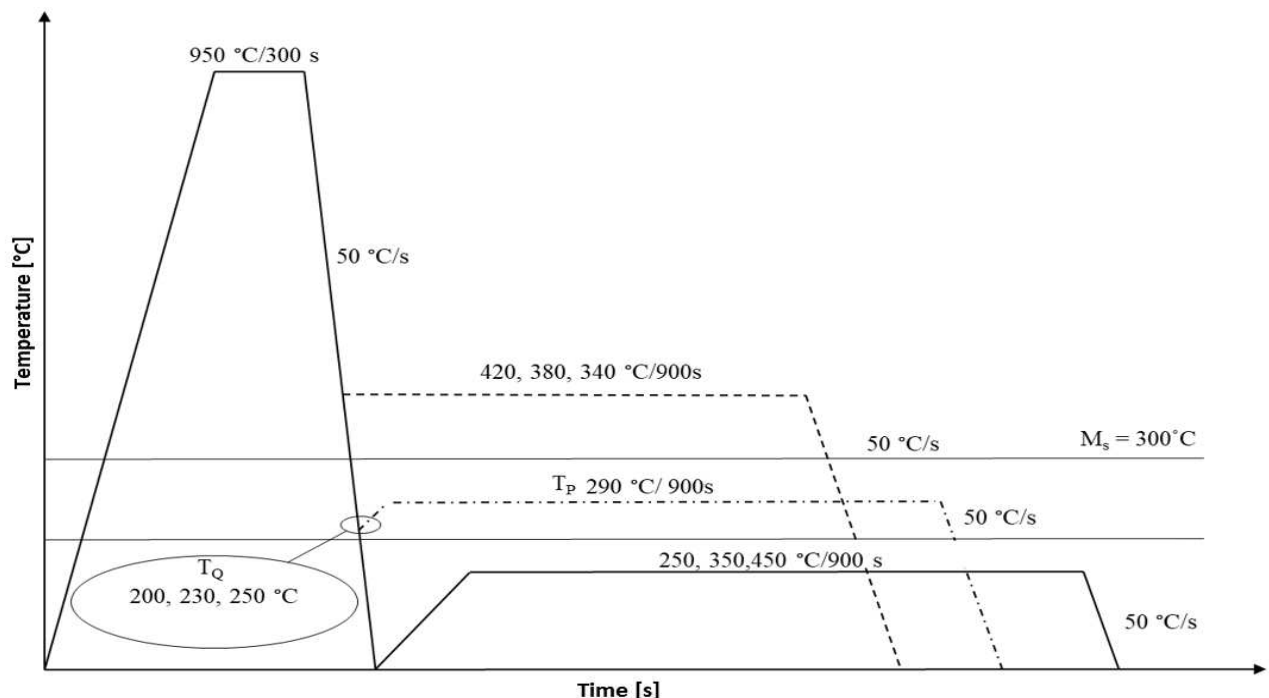
**Keywords:** Heat treatment, Steel, Martensite, Q-P process

### 1 Introduction

Treatment routes for new-generation higher-silicon forging steels lead to austenite decomposition into either bainitic ferrite, fresh martensite, and retained austenite (austempering treatment), or Q-P martensite, fresh martensite and retained austenite (Q-P processing) [1, 2]. In both cases, the resulting hardening structures are very fine and often difficult to identify and distinguish from one another – even for seasoned process engineers. The distribution of these microstructural constituents, and brittle fresh martensite in particular, dictates the final properties of finished forgings. Identification of such microstructures

is therefore important for manufacturing process design [3, 4].

Methods of microstructure identification need to be simple and effective [5]. A variety of classical techniques are used for this purpose. They do provide basic information on hardening microstructure but, unfortunately, not on the distribution or identity of individual microconstituents. Therefore, the techniques which meet the need for relatively accurate, sensitive and rapid evaluation of heat treatment of high-silicon steels are those involving colour etching. When performed correctly, colour etching methods can be highly effective.



**Fig. 1** Process profiles used for experimental specimens of 42SiCr steel

## 2 Experiments

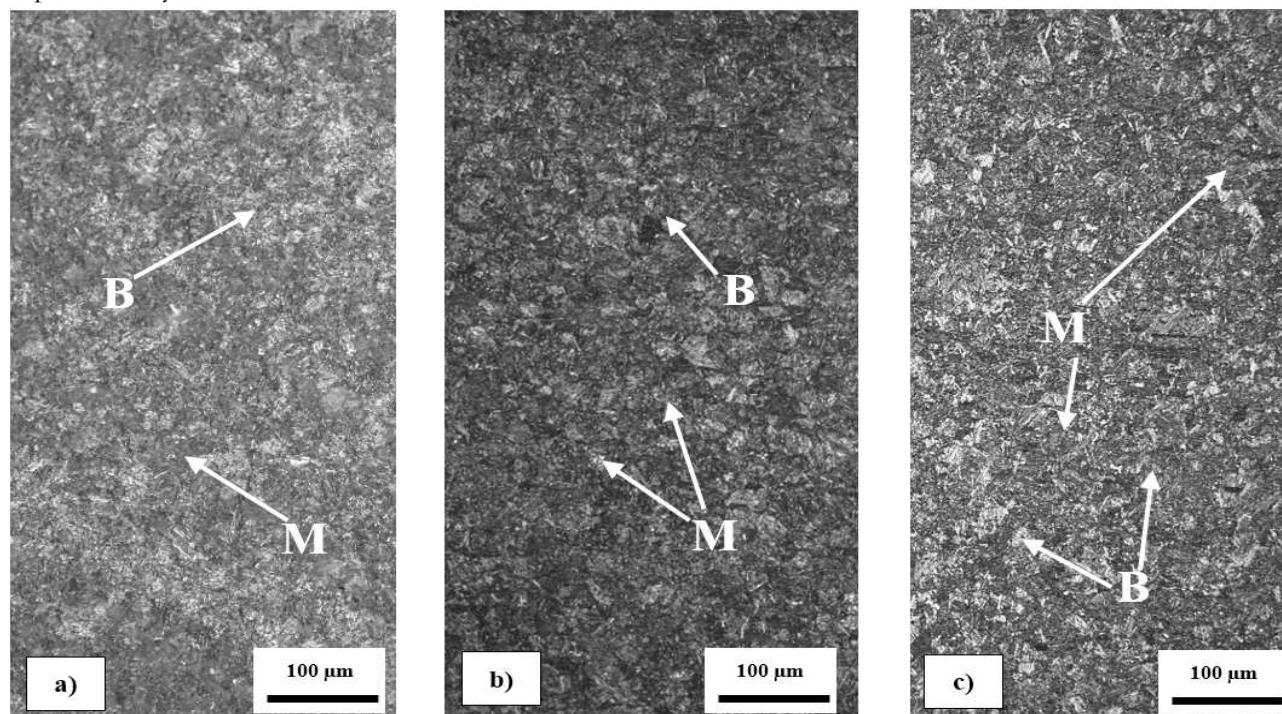
This experimental programme employed colour etching for evaluating the impact of process parameters on the microstructure of unconventional medium-carbon forging steel alloyed with 2.03% Si, 0.56% Mn and 1.33% Cr. The experimental treatment was carried out in an MTS thermomechanical simulator. Specimens were first homogenized at 1200°C for 4 hours in argon atmosphere and then cooled in air. Then they were normalized at 950°C for 1 hour and cooled in air. Once prepared in this manner, they were experimentally heat treated: austenitized at 950°C for

300 seconds, and then either austempered, or Q-P-processed, or quenched and tempered (Fig. 1)

Microstructure was revealed by colour etching with an aqueous solution of sodium metabisulfite for 8 seconds. Filters and polarized light illumination were employed to obtain the maximum contrast in micrographs. For electron microscopic observation the specimens were additionally etched with diluted hydrochloric acid.

## 3 Results and discussion

### 3.1 Microstructures of austempered specimens

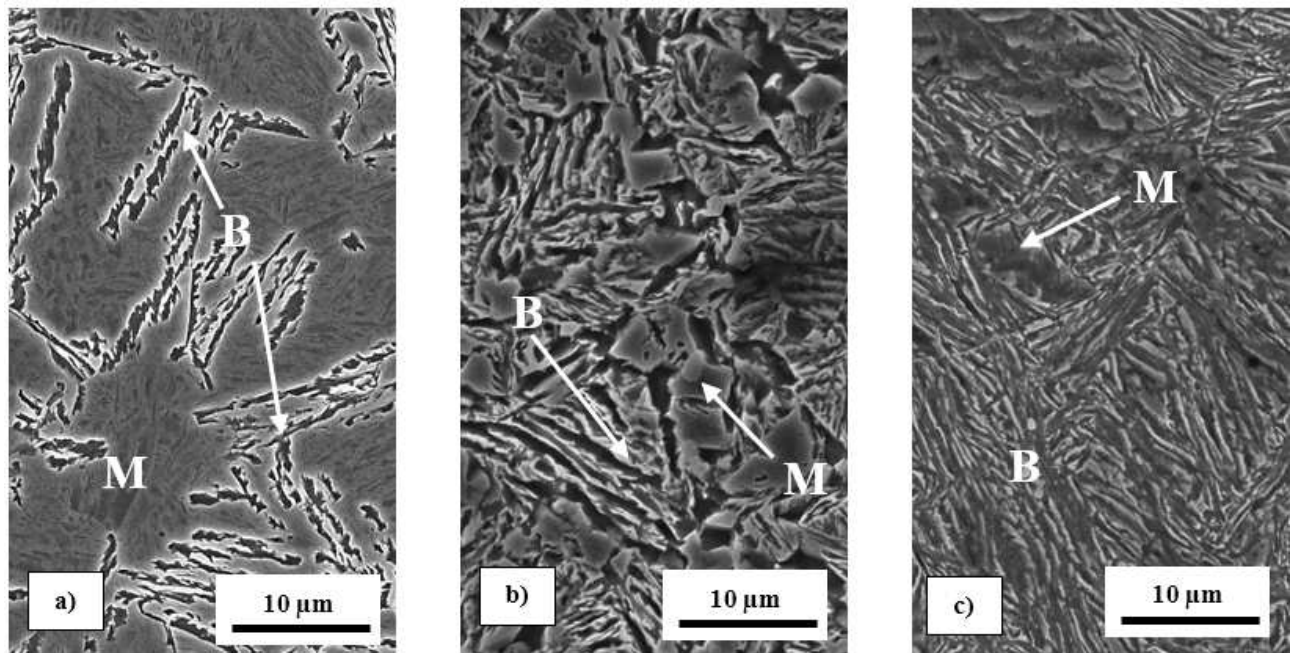


**Fig. 2** Light micrographs of austempered specimens of 42SiCr steel; M – martensite, B – bainite a) profile: 420°C/900s, b) profile: 380°C/900s, c) profile: 340°C/900s

Several structure constituents were identified in colour-etched austempered specimens using light microscopic observation (Fig. 2). To first distinguish between them and confirm their identity, their microhardness was measured. The constituent which appeared orange (light brown) showed a hardness of 700–725 HV0.05. The hardness of the blue constituent was 510–530 HV0.05. Microhardness of the constituent which appeared orange (light brown), was close to the as-quenched hardness of the experimental steel, i.e. the hardness of fresh martensite [6]. The microhardness of the blue-coloured constituent corresponded to the microhardness of bainitic structures in austempered steels with a similar level of carbon [7]. Detailed scanning electron microscopic observation confirmed the conclusions drawn from microhardness values. The microstructure of austempered specimen 420°C/900s consisted of lath martensite and needles of bainitic ferrite (Fig. 3a). The microstructure of aus-

tempered specimen 380°C/900s was formed from bainitic ferrite (major constituent) and small islands of untempered – fresh martensite (minor constituent) (Fig. 3b). Austempering conditions by specimen 340°C/900s led to formation of microstructure, which consisted predominantly of bainitic ferrite and small isolated islands of untempered martensite (Fig. 3c). Comparing to the light micrographs it was confirmed that etching with an aqueous solution of sodium metabisulfite colours the martensitic constituent orange (light brown), whereas the bainitic constituent turns dark blue.

Colour etching of austempered specimens showed big potential to reveal structural constituents – bainite and martensite according to principle of incomplete bainite transformation in silicon steels. This can be very helpful to determination of heat treatment conditions, where volume fraction of undesired fresh martensite will be depressed to minimum.

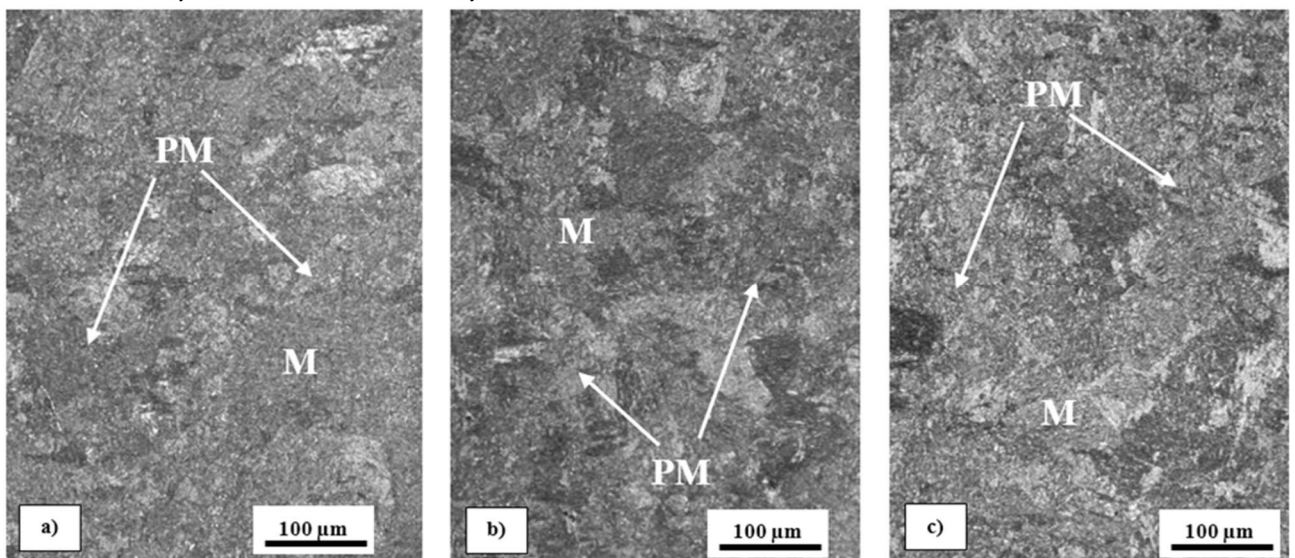


**Fig. 3** Scanning electron micrographs of austempered specimens of 42SiCr steel; M – martensite, B – bainite a) profile: 420°C/900s, b) profile: 380°C/900s, c) profile: 340°C/900s

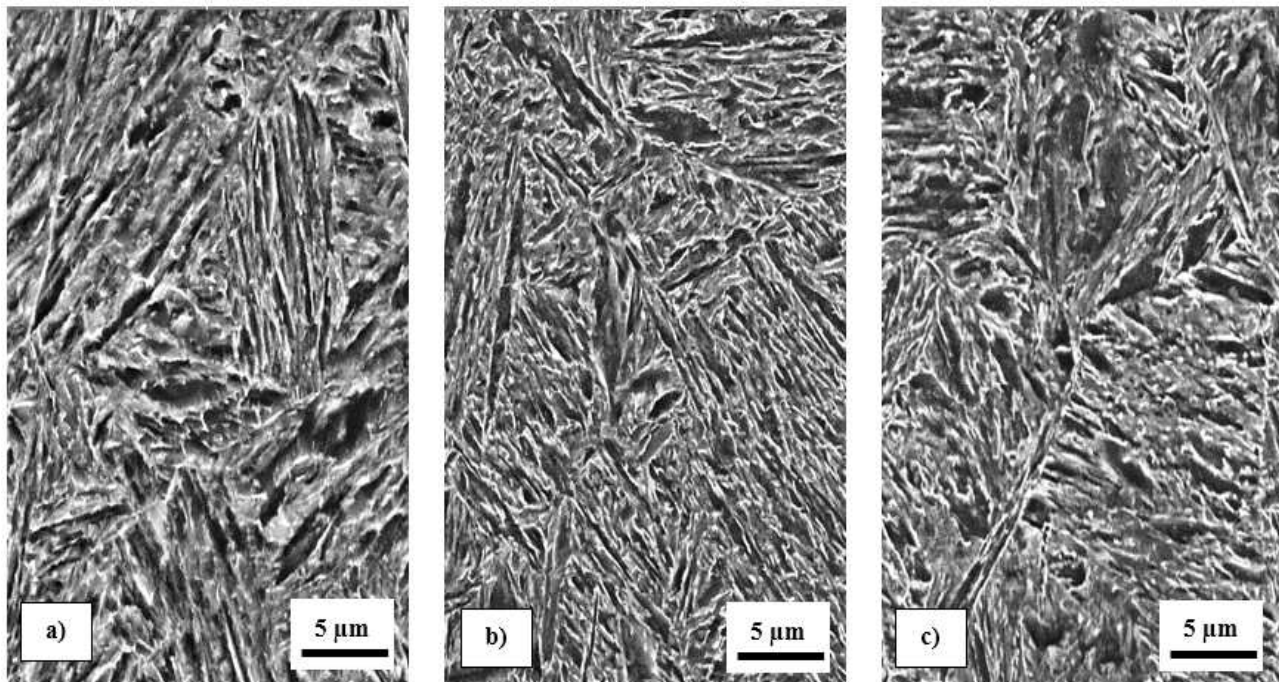
### 3.2 Microstructures of quenched and tempered specimens

In quenched and tempered specimens, colour etching clearly highlighted several constituents, which had orange (light brown), light blue color respectively (Fig. 4). The constituent with orange (light brown) color had a hardness of 700–730 HV 0.05. Taking into account the previous results, it was possible to state that the constituent with orange (light brown) color represented the untempered – fresh martensite. The constituent with light blue color was most likely tempered martensite. Several facts led to this conclusion. First - the process conditions of heat treatment during quenching from temperature of austenitization necessarily led the formation of fully martensitic

structure. During tempering some portions of martensite were pushed to the microstructural changes associated with carbon migration, formation of carbides etc. It can be expected, similar to - for example to crystallization, where to solid phase grows step by step, that tempering of martensite does not occur in whole volume at the same time but also step by step. Second - the micro-hardness of 580–640 HV0.05 corresponded with values found in partially-tempered martensite [7]. Third - the fraction of fresh martensite – orange (light brown) color decreased with increasing tempering temperatures. There is only one primary process, which can be responsible for that – microstructural changes associated with tempering of martensite.



**Fig. 4** Light micrographs of quenched and tempered specimens of 42SiCr steel – a) profile: 250°C/900s, b) profile: 350°C/900s, c) profile: 450°C/900s M – martensite, PM – tempered martensite

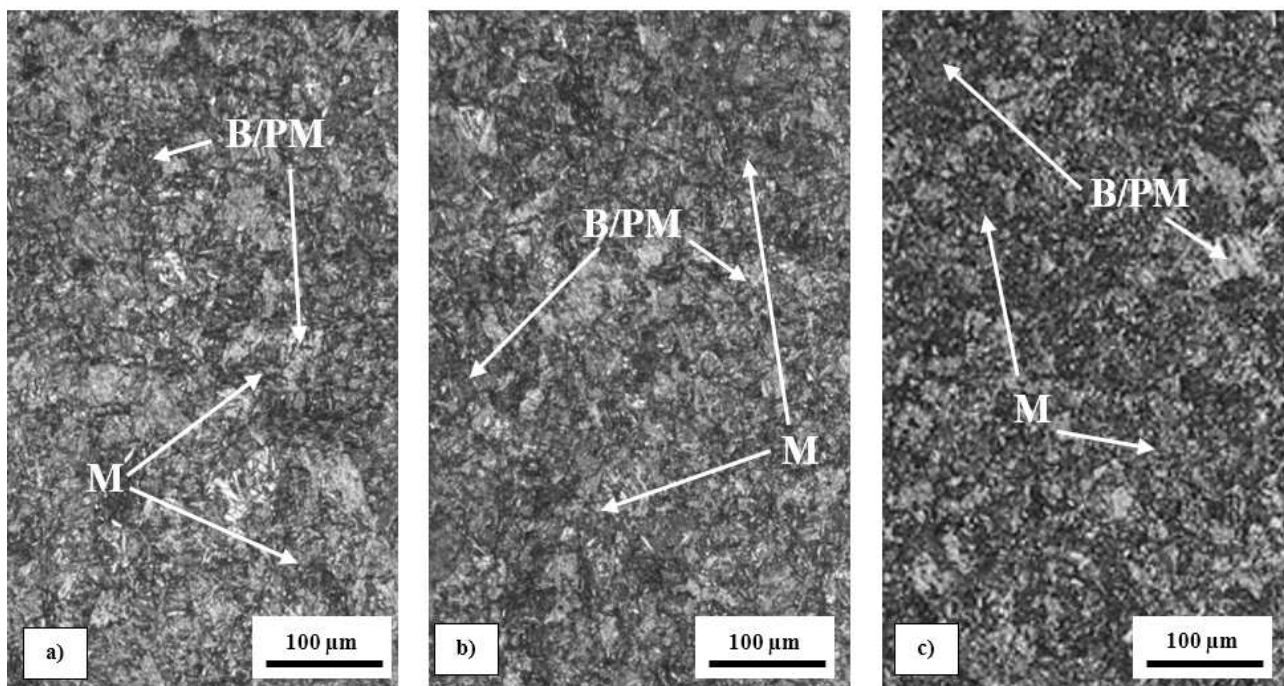


**Fig. 5** Scanning electron micrographs of quenched and tempered specimens of 42SiCr steel – a) profile: 250°C/900s, b) profile: 350°C/900s, c) profile: 450°C/900s

The facts discussed by results of light microscopy were partially confirmed by using of scanning electron microscopy (Fig. 5). Scanning electron micrographs confirmed the presence of martensitic structure in all specimens. However it was not possible to distinguish tempered and untempered martensite because of its specific morphology.

### 3.3 Microstructures of Q-P-processed specimens

Colour-etched Q-P-processed specimens contained distinguishable structure constituents whose fractions depended on process parameters (Fig. 6). The constituents had orange (light brown) and blue color. Based on the previous findings, the orange (light brown) phase, whose hardness was 700–730 HV0.05, was identified as fresh martensite. Second constituent with blue color showed hardness 510–650 HV0.05.

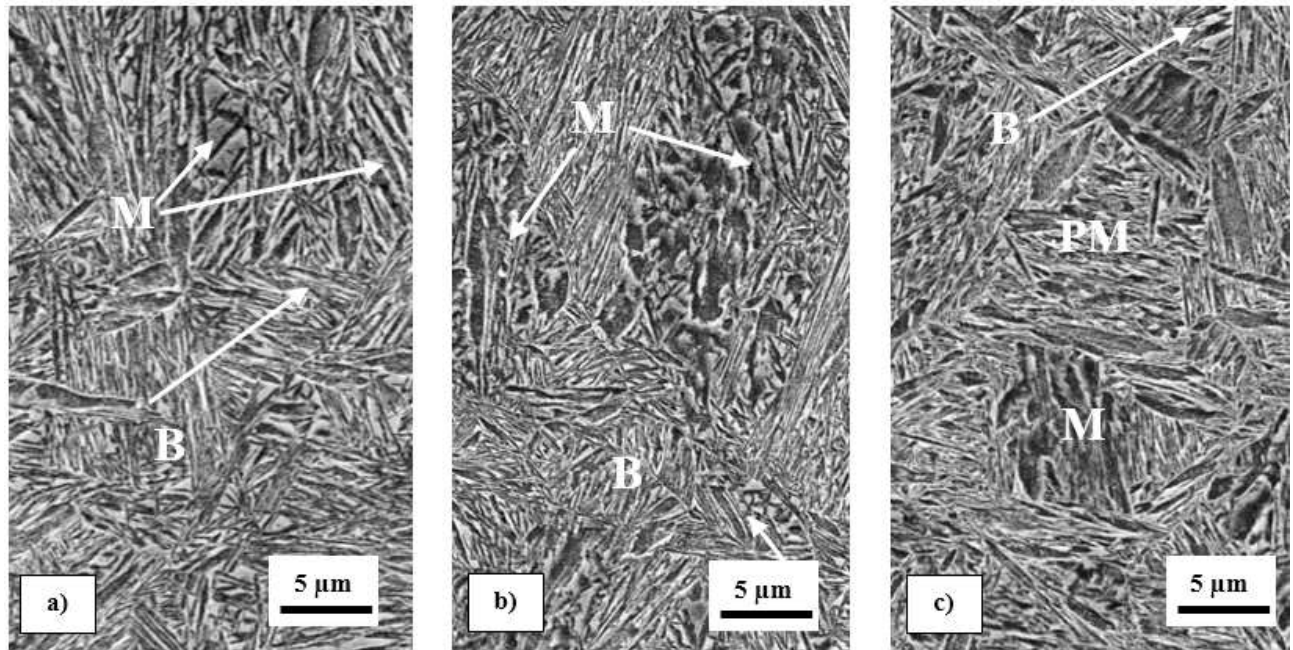


**Fig. 6** Light micrographs of Q-P-processed specimens of 42SiCr steel; M – martensite, B – bainite, PM – tempered martensite a) profile: 250-290°C/900s, b) profile: 230-290°C/900s, c) profile: 200-290°C/900s



These values of hardness were in range of both – bainitic ferrite and tempered martensite constituents. Detailed scanning electron microscopic observation of blue regions identified bainitic structure as well as tempered martensite in them (Fig. 7). The microstructure of specimen Q-P 250-290°C/900s consisted from first look from small island of un-tempered martensite and needles of bainitic ferrite (Fig. 7 a). The same conclusions were made by specimens Q-

P 230-290°C/900s, Q-P 200-290°C/900s, but different temperatures in step of quenching led to a reduction of volume fraction of bainitic ferrite, and conversely to an increase of volume fraction of tempered and untempered martensite (Fig 7 b,c). It is necessary to note, that it was not possible to distinguish the tempered and untempered martensite because of their specific morphology.



**Fig. 7** Scanning electron micrographs of Q-P-processed specimens of 42SiCr steel; M – martensite, B – bainite, a) profile: 250-290°C/900s, b) profile: 230-290°C/900s, c) profile: 200-290°C/900s

However the results showed, that during Q-P heat treatment of experimental steel the bainitic ferrite was formed. Its formation in the step of partitioning was connected with previous formation of martensite in the step of quenching. This result has significant importance for Q-P processed silicon steels because their properties can be connected with formation of carbide-free bainite rather than with redistribution of carbon from partially quenched martensite.

#### 4 Conclusion

Using colour etching with solutions of sodium metabisulfite and hydrochloric acid, hardening microstructures obtained by austempering, Q-P processing, and quenching and tempering of an unconventional higher-silicon forging steel were studied. The solution of sodium metabisulfite provided sufficient colour contrast between the structure constituents produced by the various treatment routes. The bainitic constituent was coloured dark blue, fresh martensite appeared orange (light brown), and tempered martensite had a light blue colour.

By using colour etching by the specimens proces-

sed by the austempering conditions it was easy to determine the heat treatment parameters, which led to partially or almost fully bainitic structures. This is very helpful for documentation of incomplete phenomenon of bainitic transformation in silicon steels under isothermal conditions and also for determination of the conditions under which the bainitic ferrite represents the major structural constituent.

Colour etching by the method of quenching and tempering of silicon steels can be helpful for determination of the stage of tempering. It was found that tempering of quenched martensite does not occur in the whole volume fraction, but step by step until the untempered martensite does not disappear.

Using colour etching by the Q-P processed specimens it was found, that bainitic ferrite forms in the step of partitioning. The bainitic transformation was connected with formation of martensite in the step of quenching. The presence of bainite in the structure of Q-P processed steels can be responsible for their unique properties. However sometimes the small contrast between bainite and tempered martensite does not allow to distinguish them clearly.

## Acknowledgement

*The present contribution has been prepared under project LO1502 'Development of the Regional Technological Institute' under the auspices of the National Sustainability Programme I of the Ministry of Education of the Czech Republic aimed to support research, experimental development and innovation.*

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