**Journal of Trends in the Development of Machinery and Associated Technology**
**Vol. 19, No. 1, 2015, ISSN 2303-4009 (online), p.p. 37-40**

# SCANNING ELECTRON MICROSCOPY IN ANALYSIS OF INFLUENCE OF THE ALLOYING ELEMENTS IN STEEL ON WHITE LAYER FORMATION BY HARD TURNING

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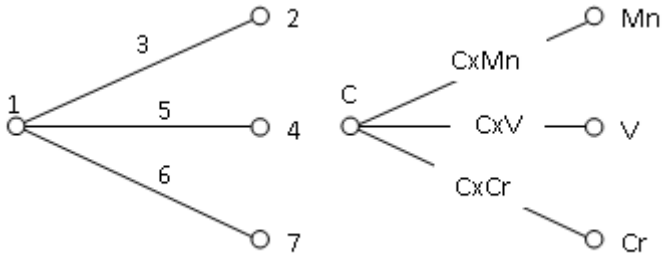
## ABSTRACT
*White Layer (WL) has been known in literature for a couple of decades. In general, the term has been used to describe non-etching, hardened surface layer of machine parts which is seen under an optical microscope as white "featureless" phase on the surface of the base material. The reason for such "metallographic" occurrence of white layer is in the fact that the same is resistant to chemical etching which is carried out in order to prepare the samples for metallographic examination. Metallographic examinations on the optical microscope are limited by wavelength of the visible light and it allowed magnifications about 1000x. In Scanning Electron Microscopy, using the electron beam instead of the visible light this magnification has been increased significantly. In this paper has been shown how the White Layer is seen under SEM microscope in comparing to optical microscopy. The feasibility of the SEM/EDS (Energy Dispersive Spectroscopy) instrumentation has been shown on example of investigating influence of the alloying elements (C, Mn, V and Cr) on WL formation in process of hard turning. It has been revealed that what has been known as a white "featureless" layer under the optical microscopy in SEM analysis has its specific microstructural form that depend on material and conditions in which the WL is formed.*
**Keywords:** Scanning Electron Microscopy, White Layer, alloying elements, hard turning

## 1. INTRODUCTION
Among the first works on White Layer were works of Babei et al. [1], whose in 1973 state that *"*Many machining, heat-treatment, and other operations carried out during the fabrication of carbon steels, alloy hardenable steels, high-strength cast irons, and other alloys produce specific structures known in the literature as white layer (or white phases, white zones, austenito-martensitic layers, "overheated skin layers", burns etc.)*"*. Golubets [2] investigated wear resistance of the white layer in relation to carbon content and concluded that "with increasing amounts of carbon in the steel the properties of the white layer change". From metallurgical aspect of view the same group of authors described WL as to be consisting of finely acicular martensite and residual austenite and containing sometimes small quantities of finely dispersed carbides. According to the character of white layer formation Griffiths [3] distinguishes three possible mechanisms of WL generation: (1) Phase transformation due to rapid heating and quenching (thermal effects); (2) Fine grain structure formation due to severe plastic deformation (mechanical effect) and (3) Reaction of the surface with the environment (chemical effect). Of course, none of these mechanisms form white layer independently but in combination with

the other two mechanisms. Most frequently used approach to induce WL formation is the hard machining [6-11]. Mehmedovic et al. [8] also defined methodology for the white layer formation on the machined surface during longitudinal turning of hardened steels. Grzesik [4] wrote "When the temperature near the machined surface exceeds the γ-α transition temperature martensite produced by friction development can form so-called white layers observed in chip micrographs". Akcan et al. [5] states "The WL is found to have a hardness of 12.85 ± 0.80 GPa, which is significantly greater than that of untempered martensite produced by various heat-treatment processes. The grain size in the WL is shown to be in the submicrometer range with values ranging, typically, between 30 and 500 nm. These two characteristics of the WL distinguish it from various structures formed in steels by heat treatment." A gross of those findings would not be possible without use of SEM, TEM, X-ray, EDS and similar advanced research equipment and technics. Ramesh et al. [6] used combination of experimental technics including Transmission Electron Microscompy (TEM), X- ray diffraction (XRD) and nano-indenation in analysis of white layer formed in hard turning. They found that white layers produced at low-to-moderate cutting speeds are in large part due to grain refinement induced by severe plastic deformation, whereas white layer formation at high cutting speeds is mainly due to thermally-driven phase transformation. Poulachon et al. [7] investigated effect of the microstructure of work material on WL formation process by means of SEM and EDS analysis. They performed the analysis on four different types of standard steel materials. One of the conclusions gained is that steels with finer microstructure develop a thicker white layer then the steels with coarser microstructure. It is well known that microstructure of steel is product of its chemical composition in combination with applied thermal treatment. Besides the carbon content, it is hard to find any other information about influence of the specific alloying elements on white layer formation. The reason might be the trend to investigate occurrences of the WL on standard type of steels as it is already mentioned afterword. In this paper is presented only part of the research related to investigation on the influence of the specific alloying elements in non-standard steel compositions on the WL characteristics by SEM analysis.

## 2. MATERIAL AND EXPERIMENTAL SETTING UP

In scope of this research, there have been produced 8 different non-standard steel compositions. The combinations are made on the basis of Taguchi $L_8(2^7)$ plan, with linear graph and controlling elements shown on figure 1. The controllable factors (alloying elements) are selected based on their "expected" way of contribution to WL formation.




| Reason for selection | |
|---|---|
| C | Basic element in steel, required in carbide formation. |
| Mn | Improve strength of material, decrease Ac1 line – austenitization. |
| V | Prevent grain growth, form carbide, improve fatigue strength. |
| Cr | Improve strength of material, form carbide. |

*Figure 1 - Linear graph for $L_8(2^7)$ orthogonal array*

The steels have been produced according to plan-matrix given in Table 3. Corresponding levels of controllable elements are presented in table 2. It is important highlight that in steel production obtaining exact level of alloying element is hardly feasible. Hence instead of exact value the practically reachable range has been defined as target value for selected elements. After producing desired combination of steels on Metallurgical Institute "Kemal Kapetanovic" in Zenica, the samples were thermally threaded in two ways. There has been defined group of quenched samples and corresponding group of tempered samples. Quenching and tempering technology were identical for all samples.In this way, not only influence of the alloying element in steels, but also their microstructure has been possible to compare.

*Table 2 - The levels –range of alloying elements*

| Level | C | Mn | V | Cr |
|---|---|---|---|---|
| 1 | 0,35-0,45 | 0,7-0,8 | 0,2-0,25 | 0,7-0,8 |
| 2 | 0,75-0,85 | 1,4-1,5 | 0,4-0,5 | 1,4-1,5 |

Machining part of the experimental research has been performed in Laboratory for metal cutting and machine tools –LORAM, of the University of Zenica. Hard turning has been performed on selected samples with worn tools (VB=0.25 mm), cutting inserts designation: CNGA 120408T grade IN22.

*Table 3. Plan-matrix of steel combinations*

| | 1 | 2 | 3 (1x2) | 4 | 5 (1x4) | 6 (1x7) | 7 |
|---|---|---|---|---|---|---|---|
| | C | Mn | CxMn | V | CxV | CxCr | Cr |
| T-1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| T-2 | 1 | 1 | 1 | 2 | 2 | 2 | 2 |
| T-3 | 1 | 2 | 2 | 1 | 1 | 2 | 2 |
| T-4 | 1 | 2 | 2 | 2 | 2 | 1 | 1 |
| T-5 | 2 | 1 | 2 | 1 | 2 | 1 | 2 |
| T-6 | 2 | 1 | 2 | 2 | 1 | 2 | 1 |
| T-7 | 2 | 2 | 1 | 1 | 2 | 2 | 1 |
| T-8 | 2 | 2 | 1 | 2 | 1 | 1 | 2 |

The tests were performed on Potisje ADA PA501M universal lathe. Cutting forces were measured by Kistler 3D dynamometer 9441B with Multichannel charge amplifier Type 5027. Metallographic preparation of samples have been finalised on MI "Kemal Kapetanovic" and on the Faculty of Metallurgy and Material Science in Zenica. Etching was performed in 2% Nital. Scanning electron microscopy investigations has been done in University Centre for Electron Microscopy of the University of Maribor. The scanning has been performed on FEI Sirion Scanning Electron Microscope (SEM), a high-resolution instrument with an electron beam.

## 3. RESULTS AND DISCUSSION

The effect of the influential factors (controllable chemical elements) on WL properties has been investigated from different aspects: thickness of the obtained WL; microhardness HV0.02 in bulk of material and in WL; content of the analysed chemical elements in bulk and WL area by EDS analysis.

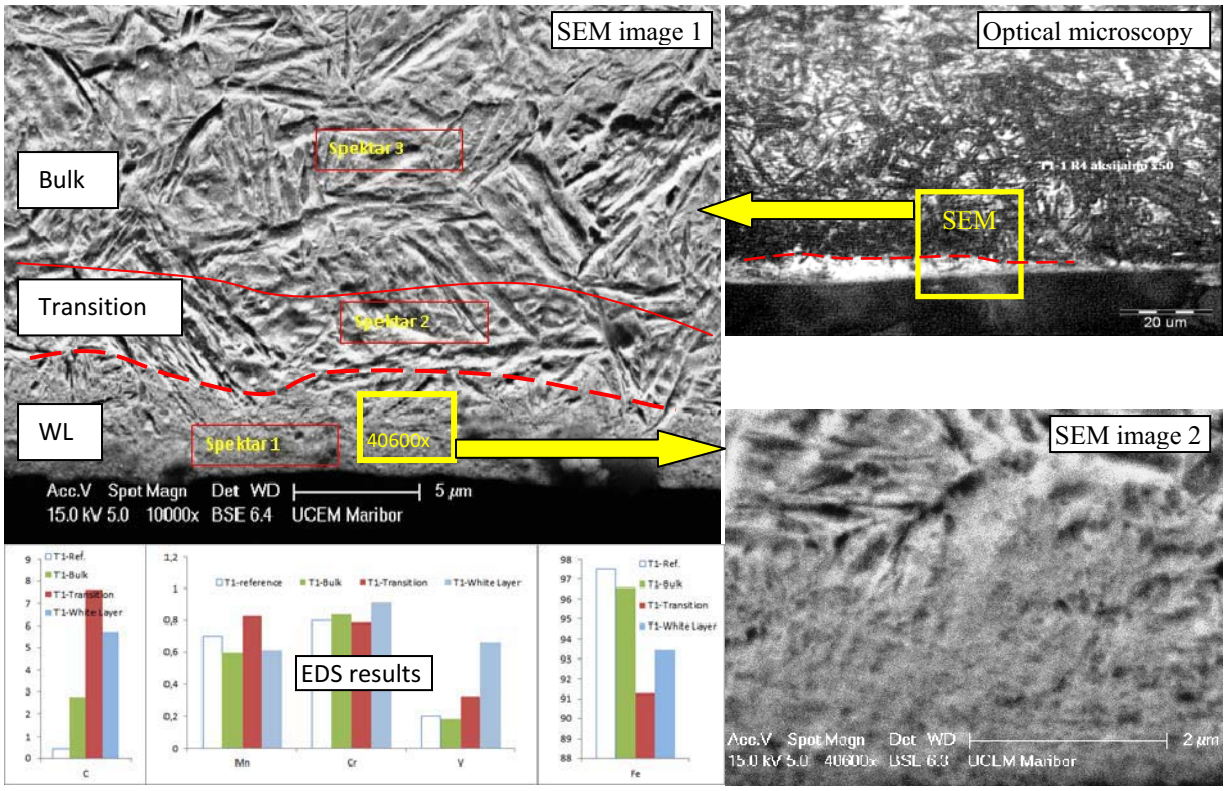



Figure 2. Comparison between SEM and optical metallography images T1-quenched

Figure 2 shows the results obtained only for one sample – T1 in quenched state. It is obvious that "unresolved" white feature on optical microscopy in SEM images became somehow "resolved" and recognised as fine crystalline structure with fine grains of nano-scale sizes. SEM image 2 confirmed that in this case the structure has a "natural" connection with bulk of the material. The surface layer seems to be exposed to remelting and rapid cooling that brought material into self-quenching state.

EDS analysis shows that content of the Vanadium increased significantly (almost 3x) in WL comparing to bulk of material. Vanadium is selected as element that prevents grain growth and its increasing in WL area goes in favour of formation fine, nano-crystalline structure. Carbon content is also increasing toward WL area. But in case of carbon is interesting that more carbon is found in transition area. This again goes in favour of statement that dark (transition) area is consisting of fine precipitates. From both of images, optical metallography and SEM images, is understendable that microstructure of the sample is untempered acicular martensitic. Martensite itself is defined as supersaturate solution of carbon in iron. During the hard machining, the temperature in narrow area arises very quickly and possibly enters the γ-area starting with austenitic transformation. Then, since the area and volume of the material is very small comparing to bulk of material quick cooling in air happened and fine martensitic structure is formed. By measuring microhardness from radial side (outside surface) it has been found that microhardness of the WL is 1600 HV0,02. From literature is known that typical martensitic structure has a micro hardness of 860 HV. Increase in micro hardness might be partially a consequence of vanadium increase since vanadium carbides are amongst the hardest one -2900 HV. In subsurface, the cooling is slower and depends on hardenability of the structure – influence of the alloying element. The carbon locked into previous martensite matrix due to higher temperature start to "escapes" from matrix and form carbides – precipitates or secondary phase.

## 4. FINAL REMARKS

Aim of the paper was to present the potential of SEM/EDS in analysis of the influence of alloying element on WL properties. An example shown in paper is related only to one sample T1 in quenched state. Similar discussion and consideration can be made for other samples too. The paper has no intention to go into detail about WL research itself but rather to highlight a way in which the SEM/EDS analysis can be used in understanding the process of WL formation. This research has just confirmed that correct use and understanding of SEM/EDS results brings significant benefits to any fundamental research process.

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