



# Crystallography, compositions, and properties of white layer by wire electrical discharge machining of nitinol shape memory alloy

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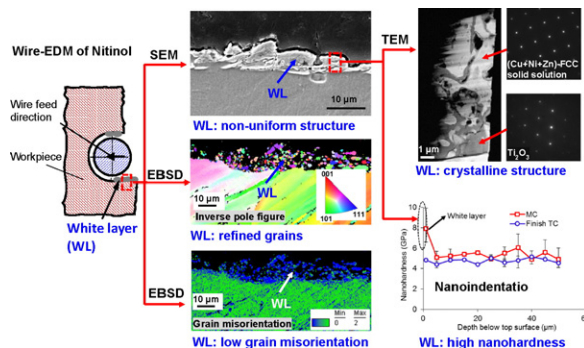
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## HIGHLIGHTS

- A thin white layer produced by EDM of Nitinol is a crystalline structure instead of amorphous solid.
- The small number of Nitinol elements and the small difference in atomic size are responsible for crystalline white layer.
- The white layer shows little crystal plastic deformation while the bulk experiences large crystal deformation.
- The white layer characterized by refined grains with random orientation is about 60% harder than the bulk.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Nitinol shape memory alloy (SMA) is very challenging to machine by conventional mechanical cutting. Wire electric discharge machining (wire-EDM) is an alternative process to machine Nitinol SMAs. The machined surface integrity is critical to product performance such as fatigue, corrosion, and wear, yet few studies have conducted a thorough investigation of the machined surface integrity, in particular white layer (WL). This work focuses on a comprehensive investigation on the crystallography, compositions, and properties of the white layer using transmission electron microscopy (TEM), X-ray diffraction (XRD), electron backscatter diffraction (EBSD), and nano-indentation. The WL by wire-EDM exhibits a porous and non-uniform bi-layered structure. The white layer of Nitinol by EDM is a crystalline structure instead of the traditionally believed amorphous solid. The upper portion of the WL consists of primarily the solid solution phase (Cu + Ni + Zn)-FCC, while Ti<sub>2</sub>O<sub>3</sub> and Nitinol austenite phases dominate the lower portion of the WL. The white layer shows less crystal plastic deformation than the bulk material, and refined grains with random orientation can be found in the WL. The nanohardness of the WL is much higher than that of the bulk material due to oxide hardening.

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## 1. Introduction

Compared to conventional mechanical cutting, wire-EDM is a competitive alternative process to machine difficult-to-cut materials including nickel-based superalloys, titanium alloys, hardened steels, conductive ceramics, and cemented carbides. The high temperature plasma

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melts and vaporizes the work material, and then the molten material is expelled by shocking pressure when the plasma channel collapses and is flushed away by the dielectric [1]. The process characteristics of contact free and force free between the wire/workpiece prevent severe tool wear and other issues inherent to mechanical cutting.

“White layer” (Fig. 1) refers to the phase-transformed recast layer formed on the machined surface. The white layer thickness may vary from 1 to 10  $\mu\text{m}$ , which depends on the level of discharge energy. In electrical discharge machining, a thin white layer (showing white color of the recast layer under microscope) is formed through re-solidification of the molten materials via rapid quenching, which results in complex phase transformation and very different microstructure from the bulk. The white layer causes great concerns in machining industry since its microstructure and mechanical properties are not well understood, but may be critical to part functions such as fatigue, corrosion, and wear.

In EDM processes, the main cut mode uses high discharge energy for rough cutting to increase material removal rate, while the trim cut mode uses low discharge energy for finish cutting to improve surface integrity. Klink et al. [2] and Li et al. [3] discovered that the white layers at the main cut mode exhibited a discontinuous and non-uniform microstructure when wire-EDM of ASP 23 tool steel and IN 718 alloys, and WLS were continuous and uniform at the finish trim cut mode. Liu et al. [4] showed that the WL thickness in wire-EDM of Nitinol SMAs can be significantly reduced at the finish trim cut mode. Klocke et al. [5] also reported that the average thickness of a white layer can be reduced using a decreased discharge energy in die-sinking EDM of AISI 4140 steel.

Kruth et al. [6] found that the white layer consisted mainly of dendritic structures which were orientated in the same direction in EDMed mold steel Impax (Uddeholm). Cusanelli et al. [7] and Klocke et al. [5] observed the similar dendritic structure as well as columnar structure in the white layer by EDM of Böhler W300 ferritic steel and AISI 4140 steel. Kruth et al. [6] detected a large volume fraction of cementite ( $\text{Fe}_3\text{C}$ ) in the white layer via XRD when EDM of mould steel Impax with the oil dielectric. Rebolet et al. [8] also found cementite ( $\text{Fe}_3\text{C}$ ) in the white layer of EDMed steel in the oil dielectric. Qin et al. [9] found a new FCC structure which was likely a complex hydride of  $(\text{Ti}_3\text{AlCr})_{1-x}\text{H}_x$  in the white layer of wire-EDMed Ti-46Al-2Cr ternary alloys in the water dielectric. Cusanelli et al. [7] found that the upper portion of the white layer consisted of residual austenite and the lower portion of the white layer was martensite in die-sinking EDM of Böhler W300 ferritic steel. Hsieh et al. [10] observed the formation of several oxides ( $\text{TiO}_2$ ,  $\text{ZrO}_2$  etc.) in the white layer when machining  $\text{Ti}_{35.5}\text{Ni}_{49.5}\text{Zr}_{15}$  in the water dielectric. Klocke et al. [11] observed the amorphous solids in the upper portion of the white layer and crystalline structures in the lower portion of the white layer close to bulk materials in wire-EDMed Vabadus 4 Extra tool steel at the finish trim cut mode.

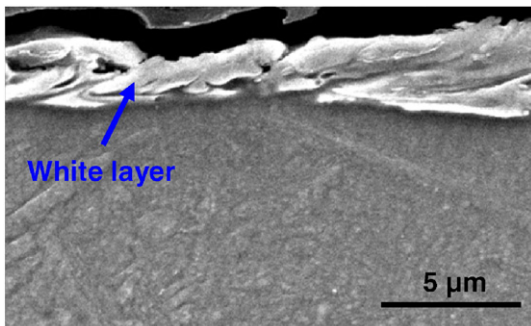


Fig. 1. White layer on the cross-section of wire-EDM NiTi sample.

The elemental compositions of the white layer may vary considerably due to the complex chemical reactions in the channel of high temperature plasma. Kruth et al. [6] detected saturated carbon in the white layer by XRD, which was diffused from the CH-oil dielectric when EDM of mold steel Impax. Hsieh et al. [10] and Li et al. [3] detected diffused oxygen in the white layer of wire-EDMed  $\text{Ti}_{35.5}\text{Ni}_{49.5}\text{Zr}_{15}$  and IN 718 in the water dielectric. Klocke et al. [12] observed that the white layer contained foreign elements (O, Cu, and Zn) from the water dielectric and brass electrode in wire-EDMed WE43 magnesium alloys at the main cut mode, while foreign elements (O, Cu, and Zn) were barely observed in the white layer at the finish trim cut mode. Guo et al. [13] and Liu et al. [4] found very little O element in the white layer when wire-EDM of Nitinol in water dielectric, but observed a trace of Cu and Zn elements (disused from the brass electrode) in the white layer at the finish cut mode. Klocke et al. [11] found that the white layer contained both foreign elements Cu and Zn (from the brass electrode) and allocated element Mo (from the carbides of the molten bulk materials) in wire-EDM of Vabadus 4 Extra tool steel at the finish trim cut mode with the oil-based dielectric.

Kruth et al. [6], Cusanelli et al. [7], and Klocke et al. [11] reported higher hardness of the white layers than the bulk in EDM of mold steel Impax, Böhler W300 ferritic steel, and Vabadus 4 Extra tool steel, respectively. Hsieh et al. [10] measured an increased hardness of the white layer in EDMed  $\text{Ti}_{35.5}\text{Ni}_{49.5}\text{Zr}_{15}$  shape memory alloys. The increased hardness of white layers was attributed to the formation of oxides and/or carbides. However, Li et al. [3] and Qu et al. [14] found the hardness of white layers were not higher than the bulk in EDM of Inconel 718 and WC-Co composites, respectively. Therefore, the hardness of white layer in EDM is material dependent. In general, the microhardness of white layer will be increased when EDM of carbon-based steels such as tool steels, while microhardness will not change or even decrease when EDM of carbon-free alloys such as Ti-/Ni-based alloys including Ti-6Al-4V and Inconel 718.

Rebello et al. [8] and Ekmekci [15] showed tensile residual stress with the maximum magnitude in the subsurface in the white layer of EDMed martensitic steel and DIN 1.2738 mold steel, respectively. However, Klink et al. [2] found the maximum tensile residual stress on the surface and sharply decreased in the subsurface when wire-EDM of ASP 23 tool steel at trim cut mode, while the maximum tensile residual stress is in the subsurface at main cut mode. It also showed that the depth of tensile residual stress is about 30  $\mu\text{m}$  at main cut mode, while it is only 5–7  $\mu\text{m}$  at finish trim cut mode. Furthermore, oil-based dielectric generally produced a much lower residual stress than that in the water-based dielectric. Antar et al. [16] showed that the tensile residual stress on the white layer surface was significantly reduced from ~570 MPa at main cut mode to ~180 MPa at finish cut mode in the wire-EDMed Udimet 720.

Although several studies have examined the morphology, microstructures, elemental compositions, hardness, and residual stress of the EDMed white layers, few studies shed light on the crystallography and properties of an EDMed white layer. A comprehensive study on the crystallography and properties of the white layer by EDM is critical. A white layer may have very different microstructures across the white layer due to the different quenching rates. There are also inconsistencies regarding the microstructure and hardness of the white layer in the literature. Furthermore, thermal damage to the grains within the heat affected zone (HAZ) is still unknown. Therefore, it is vital to clarify the crystallography and properties to understand the fundamental phenomena of the white layer.

This study will focus on the microstructures, crystallinity, and mechanical properties of white layers in wire-EDMed  $\text{Ni}_{50.8}\text{Ti}_{49.2}$  SMA. The objectives are to: (1) identify the microstructure and crystallography of the white layers; (2) evaluate the thermal effect on crystallographic evolution in HAZ from main cut mode to finish trim cut mode; and (3) study the relationship between microstructure and properties of white layers.

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