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ELECTROLYTIC POLISHING IMPACT ON TOPOGRAPHY, GEOMETRY, MICROSTRUCTURE AND THE ASSESSED DEPTH WHEN PERFORMING RESIDUAL STRESS MEASUREMENTS USING LAB-XRD AND LAYER REMOVAL

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ABSTRACT

Layer removal with electrolytical polishing is a well-established method when measuring residual stress profiles with lab-XRD. This is done to measure the depth impact from processes such as shot peening, heat-treatment or machining. Electrolytic polishing is used to minimize the influence on the inherent residual stresses of the material during layer removal. This method is done successively by material removal in incremental steps to specific depths followed by measurement. However, great control of the material removal is required. This work aims to investigate how different electrolytes and electropolishing equipment affects the resulting surface roughness, geometry, microstructure, residual stress and how to assess the obtained depth.

Comparison has been done on a ground tool steel of grade 1.1730. The results showed that electrolytical polishing with saturated salt is fastest and results in the most evenly distributed material removal but with a higher surface roughness. Perchloric acid electropolishing is slower but generates a smooth surface but may cause an increased material removal for the centre of the electropolished area. Even though, electropolishing with perchloric acid is preferable in this case as it has shown the ability to retain the stresses better than saturated salt.

Keywords: Residual stress; Profile; Electrolytical polishing; saturated salt; perchloric acid;

1. Introduction

Residual stress measurement is of great importance for the manufacturing industry to secure different manufacturing processes and for prediction and verification of components' performance in service. X-ray diffraction with lab-XRD is the most frequently used technique for stress measurement in metallic materials, which utilizes the atomic lattice as a strain gauge to calculate the stresses [1]. This technique can measure the stresses non-destructively on the surface, but for many applications, such as measuring the stress impact from shot peening of gears, machining or heat-treatment, residual stress profile measurements are required [1–3].

To perform stress profile measurements, material removal is required, which is preferably done using layer removal with electrolytical polishing [1]. Electrolytic polishing, or electropolishing (EP), is used to minimize influence on the material's inherent residual stresses during the material removal. This method is done successively by material removal in incremental steps to specific depths followed by residual stress measurements [4]. The layer

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removal utilizes an electrolyte confined in a disposer unit. When electrically activated, the anode material is dissolved causing a controlled material removal.

The electropolishing impact was studied by Alkaisee et al. who performed an investigation comparing different EP's for a shot peened steels [5]. In that work, a difference was shown between the perchloric acid and saturated salt EP that was believed to be due to difference in material removal of the different electrolytes. Deep EP and correction calculation was further discussed in the work of Pettersson et al. where residual stress profiling in induction hardened steels was performed [6].

Even though layer removal by electropolishing is a necessity, it is associated with a number of problems that may affect the resulting measured stress, as commented by Fitzpatrick et al. [1]. Hence, it is important to verify the resulting electropolished area, in terms of geometry and the topography. If the material removal is not evenly distributed, a gradual deeper material removal may be formed, which will result in a non-flat area, averaging over a depth gradient. This may then have an impact on the measured stresses, and the selection of measurement spot size, collimator, will then be very critical. Additionally, the topography of the electropolished area is also important, since a coarse surface may retain the stresses differently compared to a smooth surface. Therefore, great control of the different types of suitable electrolytes and electrolytical polishing parameters is essential.

The resulting geometry and topography after electrolytic polishing is not explored in the existing literature, which motivated this work. Therefore, investigations of the EP area have been performed, in this work, using different approaches with the target to produce a flat area with low surface roughness throughout all depths down to 150 μm . The aim has been to identify different sources of errors connected to layer removal when performing residual stress profile measurement. For that reason, electrolytic polishing of a ground steel with two commonly used equipment and electrolytes have been compared in terms of topography, geometry of the produced EP area and residual stresses. The objective has been to get a better understanding on how the layer removal differs and influences on the depth assessment.

2. Material and experimental

In this work, a commonly used tool steel for induction hardening of grade 1.1730 (C45U), was prepared by grinding a slot across the sample using a 15 mm wide grinding wheel and a depth of cut of 0.02 mm. The material has a chemical composition with low amounts of silicon, 0.4-0.6 %, manganese, 0.6-0.8 %, carbon, 0.4-0.5 %, balanced with iron and a hardness of 200HV1. The sample are 200 mm long, 60 mm wide and 30 mm thick. In the centre of the sample, a 15 mm wide slot along the sample was ground using abrasive wheel with a depth of cut of 0.02 mm. Electropolishing was performed in the centre of the slot using two equipment, Movipol-5 (equipment 1) from Struers and Couloscope CMS (equipment 2) from Fisher as seen in Fig. 1. The saturated salt electrolyte had the composition 88 g NaCl in 500 g distilled and de-ionized water. The perchloric acid electrolyte was a Struers A2 solution with the composition 70 % ethanol 10% 2-butoxyethanol, 10% distilled water and 10% perchloric acid.

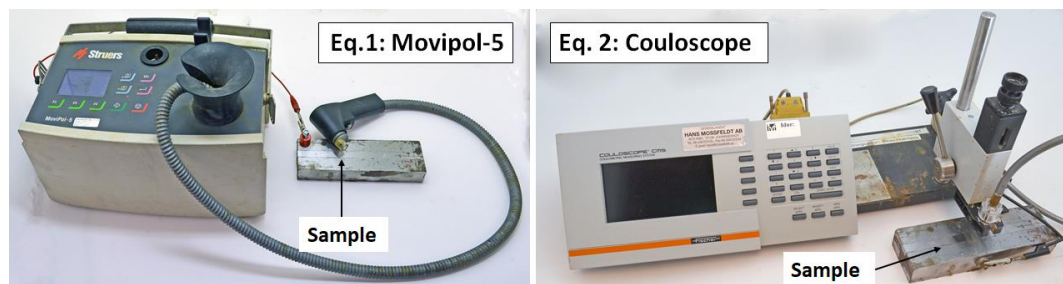


Fig. 1. Overview of the electro polishing equipment, 1: Movipol-5 and 2: Couloscope CMS.

Table 1. Process settings used for electro polishing.

Equipment	Electrolyte	Current [A]	Voltage [V]	Flow*	Material removal rate** [$\mu\text{m/s}$]
Eq. 1: Movipol-5	Saturated salt	2.8-3.0	25	6	2.12
Eq. 1: Movipol-5	Perchloric acid, A2	0.3	34	8	0.51
Eq. 2: Couloscope CMS	Saturated salt	N.A.	2.7-3.3	N.A.	0.16

*Specific setting of the equipment, ** Typical values for steel.

The geometry of the EP depth was measured using the confocal fusion technique with a Sensofar S Neox instrument. Confocal fusion is a combination acquisition method that uses both confocal and focus variation techniques [9]. The measurements were done by stitching 12x17 measurements together, each measuring 868x651 μm surface, corresponding to a total area of 6x7 mm with a lateral resolution of 1.29 μm . The depth assessments were made by a manual step height evaluation where the non-electro polished plane was selected as reference and a 2 mm diameter circular spot in the centre of the electro polished area was selected as the depth plane. The surface roughness was evaluated from measurements performed in coherence scanning interferometry mode over a selected circular area of 2 mm diameter in the centre of the circular EP area with a lateral resolution of 258 nm. The arithmetic mean height, S_a , was calculated according to ISO-25178-2:2012 to characterise the surface roughness.

The geometry was also measured for the different EP alternatives using a depth gauge. The selected measurement strategy was to measure reference positions on either side of the electropolished area and a depth measurement in the centre of the spot. The depth is then calculated by taking into account levelling of the surface. In the present work, a dial indicator depth gauge model ID F125 from Mitutoyo with a resolution of 0.001 mm was used. The depth gauge was fixated to a stand and a stable measurement table.

The residual stresses were measured using X-ray diffraction with a G2R XStress 3000 system from Stresstech Oy. The X-ray source was Chromium and measurements were performed using the modified $\sin^2\chi$ method with five tilt angles (in the interval $\pm 40^\circ$). The (211) lattice plane with diffraction angle 156.4° was measured. The stresses were calculated assuming elastic strain theory according to Hooke's law, using Young's modulus of 211 GPa and a Poisson's ratio of 0.3. Residual stress profiles were measured using layer removal with the two equipment and settings noted in Figure 1 and Table 1. All EP were done using masking by fitting a circular mask of diameter 5 mm to the EP area. The measurements were performed in accordance with the SS EN 15305:2008 in an accredited laboratory [7].

The microstructure was analysed using a Jeol LV6610 Scanning Electron Microscopy (SEM). The samples were polished in steps followed by nital (3%) etching.

3. Results

The resulting geometries, described by 3D maps and profiles across the electropolished areas, are presented in Fig. 2. The results show that the geometry changes with depth and that three different EP setups produce different geometrical features. The saturated salt profile for equipment 2 (Couloscope) is the most evenly distributed, resulting in the flattest EP-area. Equipment 1 (Movipol-5) with saturated salt shows a relatively flat distribution in the centre, but with a circular band with deeper material removal towards the outer diameter than the centre. This becomes more pronounced for greater depths. EP with equipment 1 using perchloric acid electrolyte results in a deeper material removal in the centre that becomes more pronounced for greater depths. The measured depths in these images are retrieved from the measurement by confocal fusion with the described technique in the experimental section.

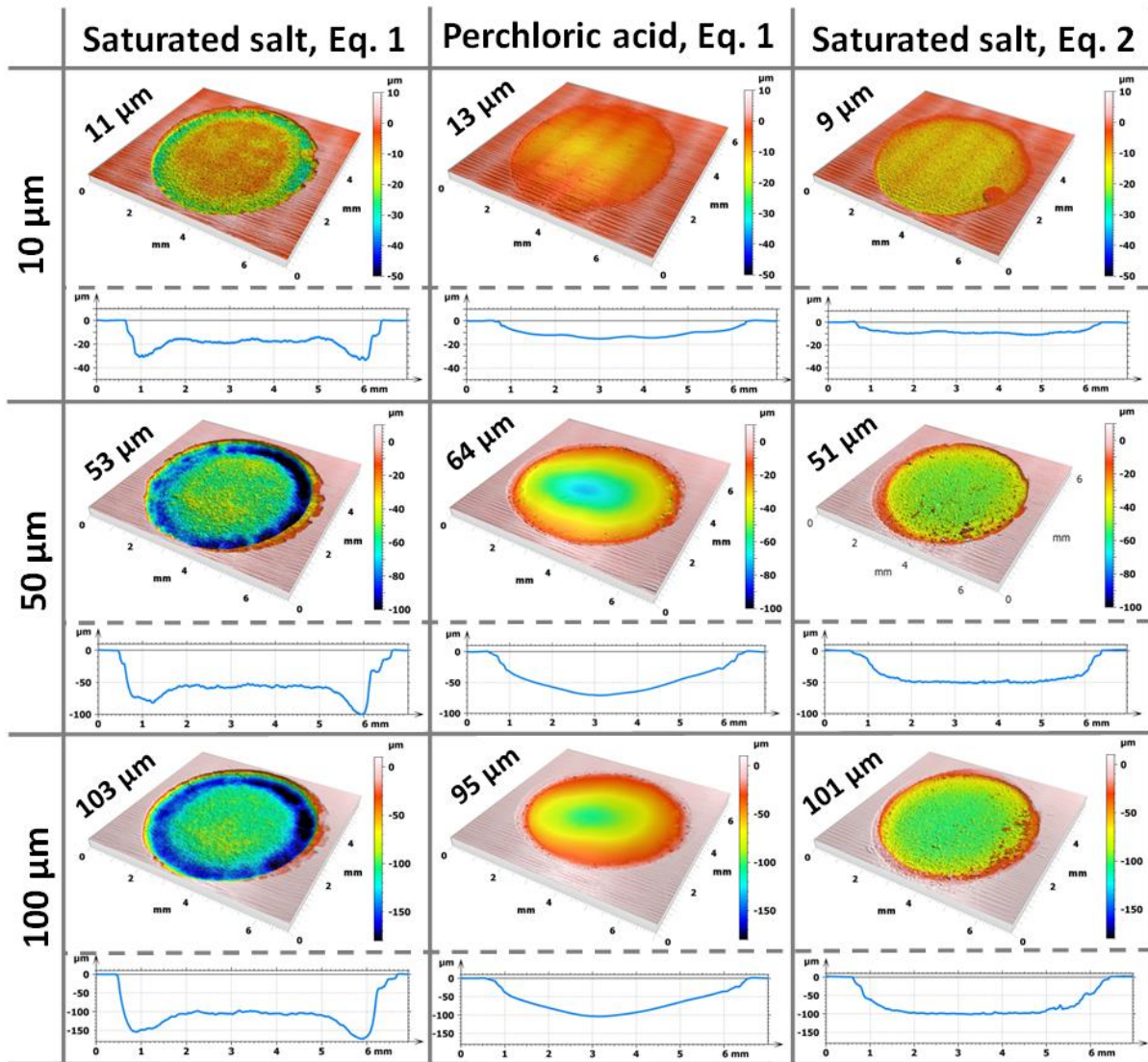


Fig. 2. 3D topography maps of the EP for equipment 1 and 2 with saturated salt and perchloric acid electrolytes.

Verification of the obtained electropolished depth is an important aspect for residual stress measurements. In Fig. 3, a comparison of depth assessments between the optical microscopy depth measurement method and the dial indicator depth gauge measurements is presented. The results show that saturated salt have almost a perfect match between the depth gauge and microscopy depth assessment while the perchloric acid, show an increasing deviation with depth. This is shown by the difference in the slope of the curves. The perchloric acid alternative show that the assessed depth is deeper for the dial indicator depth gauge. I.e., for the deepest measurement of the perchloric acid profile, the difference is 8 μm between the optical microscopy measurement method and the dial gauge indicator.

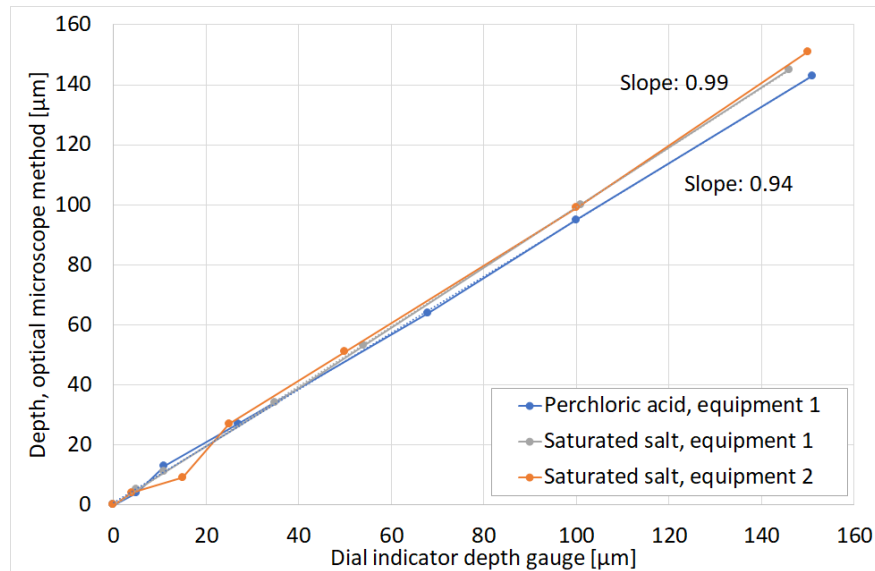


Fig. 3. Depth measurement comparison between optical microscopy method and a depth gauge for the different EP, slope from the linear trend lines.

The resulting surface roughness measured with the parameter S_a , arithmetic mean height, are shown for the different EP depths in Fig. 4 A). The initial ground surface has a S_a of $0.58 \mu\text{m}$ which changes with the different EP methods. Equipment 1 with perchloric acid is showing a decrease of surface roughness with depth while the two EP with saturated salt, results in an increased surface roughness that increases with depth. Initially, a drastic increase of S_a is shown, down to a depth of $50 \mu\text{m}$, followed by a slower increase of S_a . It is further shown that for saturated salt, equipment 1 produces a higher surface roughness compared to equipment 2.

The material removal rates are also different between the different alternatives as seen in Fig. 4 B), indicated with the slope (material removal rates) of the curves. Equipment 1 with saturated salt, has the lowest material removal rates indicated. Equipment 1 is much more efficient with higher removal rates for both saturate salt and perchloric acid. The latter is showing very high removal rates of $2.12 \mu\text{m/s}$.

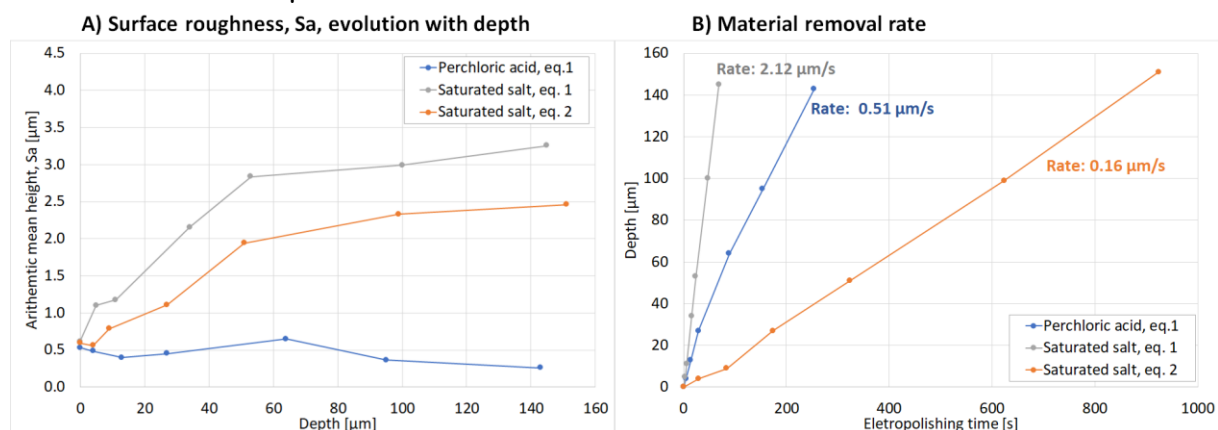


Fig. 4. A) Arithmetic mean height, S_a , evolution with depth and B) material removal rates for the equipment 1 and 2 with saturated salt and perchloric acid electrolytes.

The residual stress profiles in directions along and across the grinding direction are shown in Fig. 5. The depth is assessed based on optical microscopy depth measurement method. The results show that the two profiles produced by saturated salt EP is very similar in the surface region but have an offset of 10-20 MPa for depths greater than $10 \mu\text{m}$. The residual stress profile for perchloric acid EP show higher tensile stresses for all depths down to $100 \mu\text{m}$, after

which the three profiles coincide. All profiles show similar shape, with a drastic drop just below the surface and then gradual decrease into a core state of approximately 120 MPa. Additionally, the dial indicator depth gauge measured profile, shown as a dashed line, indicate a shift of measuring greater a depth as the EP depth increases.

The measurement across grinding direction also shows a difference between perchloric acid and saturated salt profiles. Both saturated salt profiles show a sharp decay of stresses the first 10 μm , and then a gradual decrease and the three profiles coincides at greater depths.

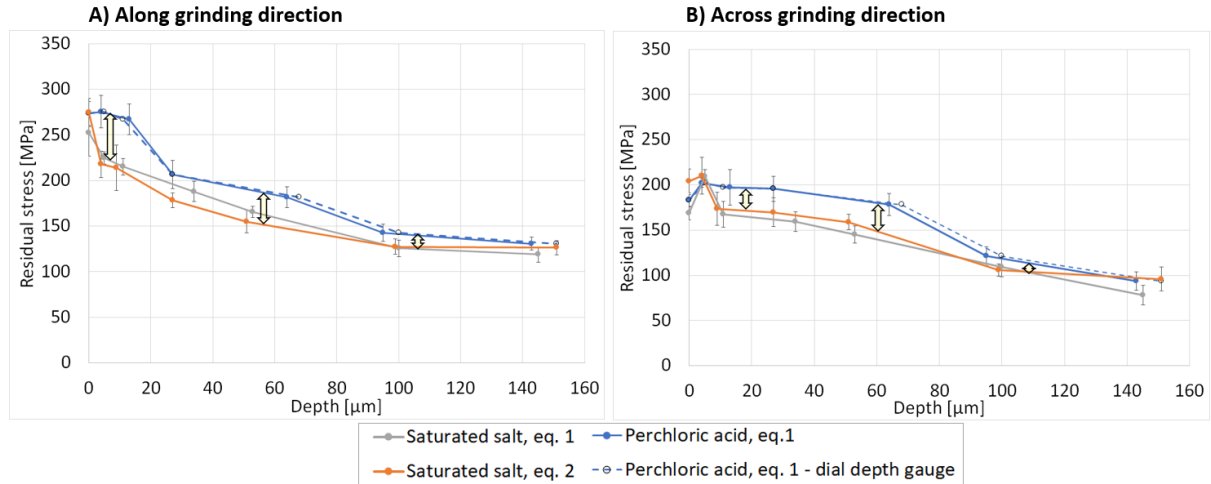


Fig. 5. Residual stress profiles in A) along and B) across the grinding direction for equipment 1 and 2 with saturated salt and perchloric acid electrolytes.

To assess measurement errors connected to the geometry of the electropolished area, a mapping of stresses was performed within the two electropolished areas produced by equipment 1. This were done by performing six measurements in a cross with an offset of 1 mm for the greatest EP depth in the profile, approximately 145 μm , for both perchloric acid and saturated salt. The results show high standard deviation values for perchloric acid electropolished area in along direction but also for saturated salt in across direction.

Table 2. Stress mapping at approx. 145 μm EP depth for equipment 1 using perchloric acid and saturated salt.

Electropolishing (EP)	Stress Along	Std. Dev.	Stress Across	Std. Dev.
	[MPa]	[MPa]	[MPa]	[MPa]
Saturated salt	53.6	3.5	58.6	9.3
Perchloric acid	40.8	15.1	35.2	5.1

The impact on the microstructure close to the surface after electropolishing were investigated using SEM as seen in Fig. 6. The material shows a mixed ferritic and pearlitic microstructure with the main constituents of Fe, Mn and Si, which was confirmed with energy dispersive analysis (EDS). The rough surface for the saturated salt EP is obvious in these images and appears to be connected to the different microstructural features in the surface. The material removal implies that the ferrite phase has resulted in higher material removal compared to the pearlite, which is protruding from the surface. The unevenly distributed material removal indicates some corrosion attacks of the sharper peaks as seen in Fig. 6 A) and B), indicated with a yellow arrow in the figure. These attacks are mainly located in the pearlite lamellar structures of the surface. However, these visual attacks are local and very shallow and could not be confirmed with EDS mapping. The perchloric acid electropolished surface is very smooth with a thin bright surface layer as shown in Fig. 6. This surface shows an evenly distributed material

removal independently of the microstructural features in the surface. EDS analysis of the surface could not detect any corrosion products.

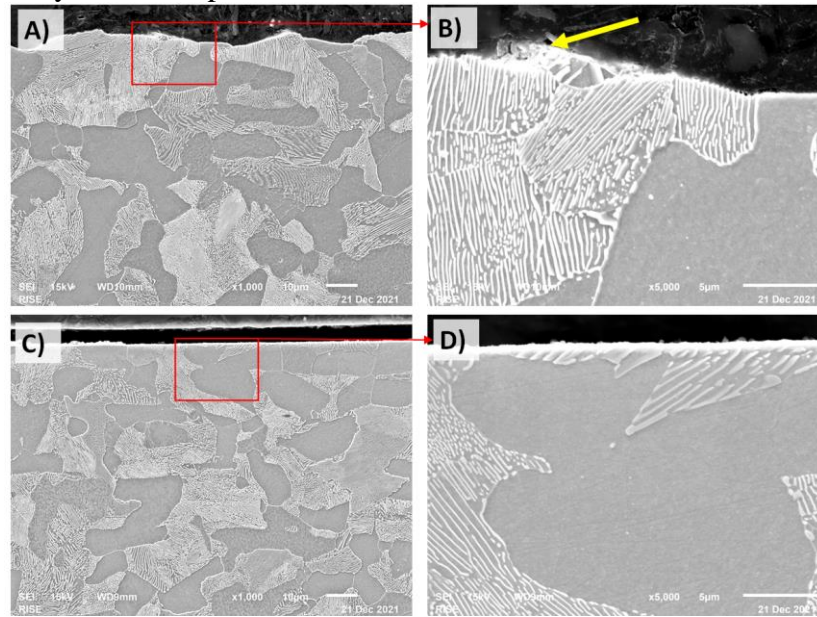


Fig. 6. SEM micrographs of surface electropolished with A-B) Saturated salt and C-D) with perchloric acid.

4. Analysis and discussion

In the present work, the impact of two commonly used EP methods has been investigated. Continued research on this topic is important since EP may result in a difference in measured residual stress as indicated by Alkaisee et al. [5]. Prior work has shown that the main contribution to the total measurement error originates from variations in the samples itself [8]. The present work has been done to increase the understanding of the errors connected to the material removal process when performing the residual stress profile measurements.

The results show that the selection of electrolyte have a great impact on the resulting topography and the distribution of the material removal of the area exposed to the EP. The two evaluated equipment show individual differences in the material removal where equipment 2 showed the best results. However, EP using saturated salt results in an increasing surface roughness with depth for both equipment. EP with perchloric acid, on the other hand shows a much smoother surface, but an accelerated material removal for the centre part of the EP area. This results in a deeper pit in the centre, which will have an influence on the measured depth for the profile. It will also have an effect on the measured stresses since the measurement is averaging over different depths. I.e., the greatest depth in the centre of a 2 mm circular area, equal to the XRD measurement area, is 150 μm while the average depth in the entire 2 mm circular area is 143 μm . In this case, this depth assessment difference did not show a large impact on the resulting stress profile since the stress gradient at these depths is low. Also, the stress mapping in the EP area had large standard variation for both EP alternatives implying variations from the material itself rather than from the EP. However, it could be critical for a stress profile that has a large stress gradient, like a shot peened surface. This result also highlights the importance of high accuracy of the EP depth measurements, especially if the sample surface is complex, non-flat and requires perchloric acid EP. The EP depth is normally measured using a depth gauge across the EP area. However, if the sample is difficult to align, only the centre of the area is used for the depth measurement, then the depth should then be calculated using corrections for the misalignment. In this work, a method using optical topography measurements by averaging over the EP area that will be measured by XRD, has instead been proposed when measuring the obtained EP depth.

The resulting residual stress profiles for the two equipment and electrolytes showed a clear difference where the perchloric acid profile had a higher tensile stress compared to the saturated salt profiles. The difference for the two saturated salt profiles is small. The reason for the difference in residual stresses between perchloric acid and saturated salt is related to the topography and the geometry of the electro polished area but may also be and affected of stress relaxation from corrosion for the saturated salt polishing, also indicated by Alkaisee et al. [5]. This implies that perchloric acid EP is less harmful in this perspective, which may retain the stresses better compared the saturated salt EP. However, development of perchloric acid EP is required in order to improve geometry of the pit-shaped spot. This would include adjustment of the flow and concentration further suggested by Fitzpatrick et al. [1].

To summarise, the present work shows the importance of being aware of the limitations of the layer removal technique to be used. The saturated salt EP is a fast and efficient method but may cause corrosion attacks on different microstructural features. The perchloric acid EP is more time consuming and hazardous to handle but result in a smoother surface, which is more prone to retain the stresses. Therefore, for this type of non-hardened materials it is suggested use perchloric acid but with a risk of uneven material removal which is important to evaluate.

5. Conclusions

The following conclusions have been drawn:

- The selection of electrolyte has an impact on the residual stress shown as the profiles produced with electropolishing using perchloric acid electrolyte, had higher tensile stresses throughout the complete profile, implying a greater ability to retain the stresses.
- Electropolishing with saturated salt electrolytes result in higher surface roughness and indications of corrosion attacks on the pearlitic constituents in the surface microstructure while perchloric acid resulted in a smoother surface without corrosion attacks, both of which may affect the ability to retain the stresses.
- Both perchloric acid and saturated salt electropolishing may result in non-evenly material removal, which requires profilometry measurements to assess the depth correctly. This could be very critical for components with high stress gradients at greater depths.

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