NON-DESTRUCTIVE METALLOGRAPHIC ANALYSIS OF SURFACES AND MICROSTRUCTURES BY MEANS OF REPLICAS

D. Zuljan, J. Grum

University of Ljubljana, Faculty of Mechanical Engineering, Aškerčeva 6, 1000 Ljubljana, Slovenia,
E-mails: darjo.zuljan@fs.uni-lj.si, janez.grum@fs.uni-lj.si

ABSTRACT

The paper treats the application of a system of producing replica to non-destructive metallography and non-destructive analysis of surface topography. The replicas were produced by the Struers company and are known under trade name of RepliSet-F5. They are made of fast-curing two-part silicon rubber capable of flexible, high-resolution capturing of a surface image. A new type of replicas was applied to the surface analysis. This type is designed for general purposes, particularly for flat surfaces, at normal and elevated temperatures. Some older replicas of the same firm named TRANSKOPY were also applied. These ones are reflective metallographic replicas for convex and concave part surfaces applied to non-destructive testing. Both systems of the replicas were applied to a microstructure analysis, crack analysis of axles and straightening rolls. The results obtained in the analyses of the microstructure, surfaces, damages, and cracks show that the replica technique can be exceptionally efficient particularly in those cases where on-site facilities and usually also large and expensive facilities in operation, for which periodic examinations are specified, are analysed.

Keywords: RepliSet-F5 system, Transcopy replicas, Optical microscopy, Cracks, Damages

1. Introduction

Non-destructive metallography of surfaces makes it possible to analyse a material microstructure and a surface condition as well as various surface damages occurring due to overloading or improper tribological circumstances. Machine or tool parts inspected are not damaged during the surface preparation. It is also required that the replica applied is strong and elastic enough not to get damaged when removed. As the replica does not damage the machine part and does not chemically affect its condition, the machine part can further operate if the inspection performed confirms its quality. The way of preparing the machine part, i.e., the area to be inspected depends on the requirements or objectives of the examination to be performed and on the accessibility of the area to be inspected. The inspection of the surface condition or microstructure can be performed in two ways:
If the part to be examined and its environment provide enough room for the surface preparation and observation, then the surface inspection can be performed directly with an optical microscope. For this purpose adapted optical microscopes are available.

In the opposite case, a replica is produced, which is then observed with a light or electronic microscope in a laboratory.

The optical microscope makes it possible to observe the replica in bright or dark field. The area at the machine part or the tool part to be examined shall be prepared with a suitable preparatory technique which is the same as the usual surface preparation for the optical microscopy [1]. The area concerned shall be first mechanically treated by grinding and/or polishing or electrolytic polishing, then it shall be etched so that the microstructure of the surface becomes uncovered. The surface preparation is similar to that used in the general metallographic analysis with the optical microscope. In case the machine part has already been fine-ground or polished because of functional requirements, it is needed to etch it for the microstructure analysis. It is different, however, with the analysis of the surface condition due to tribological conditions, in which case the surface shall only be cleaned and a replica shall be made for subsequent observation.

Electropolishing and etching can be carried out with the MOVIPOL, a portable unit produced by Struers. Fig. 1 shows two portable units of Struers, i.e., the TRANSPOL designed for the preparation of the area to be examined by grinding (Fig. a) and the MOVIPOL designed for etching (Fig. b). This is followed by the production of a replica and finally by the observation of the microstructure with the optical or electronic microscope.

![Fig. 1: Machines for surface preparation for non-destructive metallography: TRANSPOL (a) and MOVIPOL (b).](image-url)

For an analysis of topographical features of the surface of a machine or tool part the surface shall be thoroughly cleaned, without any preliminary mechanical treatment. The preparation of both simple and exacting surfaces may be made easier with the application of admissible chemical media for the elimination of colour, grease, and other impurities. One finds commercially available various types of replicas made of different materials. The materials available are practical for application and permit the production of a replica of the surface concerned in a few minutes. One finds commercially available also metal-coated plastic foils prepared in advance for the production of replicas. The metal coating provides efficient light reflection when the replica surface is inspected with the optical microscope. This also provides a higher quality of the surface image, and additional mechanical hardening of the replica results in easier and safer handling. Consequently, the modern replicas are made of two-part silicon rubber. The latter is squeezed from an adapted gun with a piston to the area to be examined. The surface preparation
technique requires moderate to deep etching of the surface to be examined in order to obtain a well-profiled replica for a good and efficient topographical analysis of the surface. In addition to the employment of a mass for replicas, the employment of backing paper is also recommended. It allows the production of an even thinner replica. Such a replica is then stuck, with a double-sided adhesive tape, to a flat glass slide, which enables subsequent high-quality observation of the surface with the optical microscope. The backing paper makes it possible to produce thin replicas of particularly exacting surfaces, which can be analysed with the optical microscope. The replicas produced with the Struers backing paper can also be shadowed and subsequently analysed with a scanning electron microscope.

Figure 2 shows the components of the RepliSet-F5 unit for the production of replicas. The RepliSet-F5 system consists of a dosing pistol with a piston for squeezing-out, a container with two-part silicon rubber, and a mixing nozzle for the application of the replica mass to the surface to be analysed at the machine part. The mixing nozzle shall be replaced after each application because rests of the mass remain in the nozzle, become hard, and block the nozzle. For the production of a quality replica it is important to know the temperature of the surface of which a replica is to be made. Silicon rubber can be applied to a surface of machine parts both standing still and operating in a temperature range from -10 to 180 °C.

The procedure of making a replica with the Repli-Set-F5 system is following:
1. The surface of the machine part concerned shall be cleaned and degreased. Degreasing is usually accomplished with ethyl alcohol, similarly as in the common optical microscopy for the observation of the surface topography.
2. When the condition of a surface or a crack reaching to the surface is to be observed, cleaning of the surface with acetone is recommended. Irrespective which cleaning medium is used, its remnants shall be carefully and thoroughly removed from the surface so as not to react with silicon rubber used in making the replica.
3. With exacting microstructures, surfaces or cracks, the cleaning medium shall be additionally rinsed and, as circumstances require, the surface dried with hot air.
4. Then follows the application of the replica mass capable of hardening, i.e. having favourable strength and elastic properties, and thus being capable of efficient removal from the surface.
5. Good preparation of the surface, a quality mass, and careful making of the replica result in high-quality production and observation of the replica with the optical or electron microscope.

Figure 3 shows the application of two-part silicon rubber to the surface and removal of the replica from the surface concerned.

Fig. 2: RepliSet-F5.
The RepliSet F and T replicas are suitable for the observation with the optical microscope with low magnifications even at very high magnifications, e.g. x500. The replica shows such properties that light is reflected from the surface in the same way as in common observation of a metal specimen. The RepliSet-F5 system makes it possible to make a replica with high accuracy ensuring a minor deviation of size of 0.1 µm, which strongly exceeds the resolving power of the optical microscope. It is very important that the nowadays replicas show a stable shape immediately after being removed from the specimen surface as well as during the observation with the microscope. This is of major importance for an efficient microstructure analysis of the surface material, analysis of surface topography, and analysis of surface damages. Dried replicas have a thickness of some millimetres and should be well-removable from a damaged surface with a crack or any other type of defect. The topographical analysis of the surface can be accomplished efficiently also with various contact or contactless measuring techniques such as laser technique, a measuring projector for 2D or 3D observation of damaged surfaces, and others. The replicas are suitable for the 3D topographical analysis with the scanning electron microscope. They can also be shadowed to improve the assessment of wear, damage or crack at the surface. In the process of making a positive replica or a two-stage replica it is necessary to dry the single-stage replica completely, which may take even up to 24 hours. Then the two-component mass for making the positive replica is applied. The positive replica obtained in this way shall be removed from the surface or the single-stage replica only after the completion of drying. The drying time depends on the type of replica and is selected, on the recommendation of the manufacturer, from relevant tables.

Fig. 4: Micrograph of pitting damage obtained with electron microscope.
Figure 4 shows a micrograph of a replica with a surface damage in the form of a pit detected by the scanning electron microscope.

The non-destructive testing method using the replica technique is applied to the control of different parts of various power devices. This is an indirect observation of the momentary microstructure in periodic inspections to assess the material surface and subsurface conditions of the part concerned resulting from a long-term operation at elevated temperatures. Thus hidden thermal overheating in the system in the period between two subsequent periodic inspections can be controlled. This makes it possible to efficiently predict the time of further safe operation of the system or its individual elements. The methodology of non-destructive metallography is applicable to studies of the microstructure of parts of different devices and contributes to diagnosing the causes of damages, determining the type and condition of the material, and to assessing the surface condition with reference to the technology of production, material treatment or operating conditions in the system, which show a more or less explicit influence on the microstructural changes.

2. Experimental procedure and results of the analysis

The paper treats the application of the non-destructive metallography to the investigation of cracks at straightening rolls. The straightening rolls are used in plate-straightening machines. Non-destructive methods are highly suitable for detecting cracks at the surface of the straightening rolls. It is with the visual examination, penetrant examination and/or magnetic particle examination that the presence of the cracks can be confirmed, and the condition of the surface or material, i.e. the quality of the element to operate in a device or a machine, assessed. The information obtained makes it possible to efficiently describe the condition of the material, i.e. of a device, and permit to make decisions as to the repair of damages. For a non-destructive analysis of the surface topography, damages or cracks at the surface, it is the Struers RepliSet F system that is highly suitable. For concave and convex surfaces of machine or tool parts, the TRANSKOPY, Struers reflective metallographic replicas, are highly suitable. Both variants of producing replicas for the surface analysis are complementing each other and are a high-quality accessory in research work as well in the practical material inspection.

The micrographs of the replica surfaces were made at an Leitz-ORTHOPLAN microscope with an ORTHOMAT photo-camera and Leitz objectives with the illumination in bright field. The micrographs were shot with a professional digital camera, NIKON COOLPIX 5400, equipped with additional optics permitting mounting of the camera into the microscope.

![Image of axle with crack (a) and image of surface of replica with crack (b).](image_url)
Figure 5 shows an axle with a crack (Fig. 5a) and an image of a replica made with the optical microscope with the illumination in bright field (Fig. 5b). The crack occurred after induction hardening of the axle because the hardening parameters were not chosen properly. Treating of a crack at cylindrical or spherical surfaces is somewhat more difficult due to the necessary adaptation of the optical system to the bent surface with the crack. The thin RepliSet-F5 replica for the original surface permits easier and more reliable observation of the crack. The thin replica was stuck to the flat glass slide, which permitted good and reliable observation and analysis of the surface with the optical microscope as well as durable filing of the replicas. Figures 6a and 6b show an image of replicas of a crack and pitting of the surface, the replicas being illuminated in dark field. Pits in the surface may be due to the corrosion processes during the operation of the machine part.

Fig. 6: Representation of crack by means of replica made, with exposure in dark field (a) and replica of surface pits (b) with illumination in dark field with optical microscope.

Fig. 7: Photos of straightening roll.

Figure 7 shows a photo of a straightening-machine roll for straightening of plate in coil. The straightening roll was made of heat-treatment steel, its core was in the heat-treated state and had an induction-treated surface. The straightening roll was then provided with a hard chromium coating. It was as early as after a week's time that the roll became locally damaged, i.e. a section of the chromium-coating exfoliated. A detailed inspection showed web-like cracks across the entire roll surface. The observation of the straightening roll with the unaided eye showed web-like cracks at the chromium-coated surface. A decision was taken to make replicas of the surface condition and make photos of the surface at different magnifications. Images of the replicas were made with a microscope, a product of CARL ZEISS from Jena, and showed the cracks at the surface of the straightening rolls. For the analysis the illumination in dark field was used. For
documenting a digital camera mounted at the microscope with a suitable adapter and adjusted to microscope optics was used. Figure 8 shows images of replica surfaces at different magnifications. The replicas were made at the straightening machine. They were an older version of Struers replicas known under trade name of TRANSCOPY and as metallographic reflective replicas. The images show light web-like cracks on the dark chromium coating as a background, which allows very reliable measurement of crack size and makes the assessment of causes for the crack occurrence at the thin surface layer easier.

TRANSCOPY (Struers) - metallographic reflective replica

![Images of replica surface of straightening roll with hard chromium coating obtained with optical microscope, illumination in dark field, at magnifications given: x10 (a), x16 (b), and x25 (c).]

Fig. 8: Images of replica surface of straightening roll with hard chromium coating obtained with optical microscope, illumination in dark field, at magnifications given: x10 (a), x16 (b), and x25 (c).

Figure 9 shows an image of the TRANSCOPY replica used, which is suitable for the study of the surface of the straightening roll with the illumination in bright field.

![Image of surface of metallographic reflective replica TRANSCOPY of straightening roll with hard chromium coating.]

Fig. 9: Image of surface of metallographic reflective replica TRANSCOPY of straightening roll with hard chromium coating.

Figure 10 shows web-like cracks on the hard chromium coating made visible with the magnetic particle examination under ultraviolet light. The known surface preparation for the magnetic particle examination with magnetizing and surface illumination resulted in a very strong emission at the crack locations, where the fluorescent magnetic particles got particularly concentrated. The picture was taken with a NIKON camera and the surface was illuminated with ultraviolet light. Thus the initially slightly visible cracks at the surface were made contrasting and clearly visible with unaided eye or a magnifying lens and a microscope. This is particularly
important when automatic measurements of crack size are performed or a very convincing image is required to give proof of something or for filing purposes.

Fig. 10: Web-like cracks at chromium-coated surface of straightening roll after magnetic particle testing at lower (a) and higher magnification (b).

The micrographs were produced with the ORTHOPLAN-Leitz optical microscope. The photos were produced with the digital camera equipped with additional optics. Figure 11 shows two micrographs of the hard chromium coating at a magnification of x100 and x200 respectively. It can be inferred from the micrographs that the chromium coating is rather thick but unfortunately also porous. By means of the Image Tool V.3 programme environment it was found that the volume porosity amounted to approximately 5.5% of the size of the entire coating. The mean value of the thicknesses of the hard chromium coating was 0.24 mm.

![Micrographs of hard chromium coating. 100 x and 200 x.](image)

The hardness of the straightening-roll surface was measured with a ZWICK 3212 hardness tester, which makes it possible to measure Vickers microhardness with weaker loads. It was decided to measure microhardness because the chromium coating at the surface was very hard and thin and because the joint between the chromium layer and the substrate was weak. The force of indentation was chosen with regard to the mode of microhardness measurement at hard and thin surfaces. In the present case the tip load amounted to 2.0 daN. For micorhardness
measurements points chosen at random along the roll and at the roll circumference were chosen (Fig. 12). The mean value of hardness at the straightening-roll surface at three points and at four positions is given in Table 1. The average microhardness value at the straightening-roll surface amounted to 912 HV, which was the hardness of the chromium coating.

![Fig. 12: Cross section of straightening roll showing microhardness measuring locations and positions for surface of hard chromium layer.](image)

Table 1: Microhardness at surface of hard chromium layer at straightening roll.

<table>
<thead>
<tr>
<th>POINT MEASUREMENTS</th>
<th>HARDNESS VICKERS HV</th>
<th>2,0</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>POINT 1 HV 1,0</td>
<td>POINT 2 HV 2,0</td>
</tr>
<tr>
<td>Position 1</td>
<td>949</td>
<td>891</td>
</tr>
<tr>
<td>Position 2</td>
<td>920</td>
<td>920</td>
</tr>
<tr>
<td>Position 3</td>
<td>891</td>
<td>891</td>
</tr>
<tr>
<td>Position 4</td>
<td>864</td>
<td>980</td>
</tr>
</tbody>
</table>

3. Conclusions

- Non-destructive metallography using replicas developed from an experimental technique at a transmission electron microscope and is a quite indispensable technique in monitoring of material condition during thermomechanical loading of individual components of power systems.
- The visual inspection of the straightening-roll surface showed that the surface was cracked; therefore additional microscopic inspection of the roll surface at a magnification of some ten times was performed. The microscopic inspection of the surface using replicas confirmed that there were very fine cracks at the straightening-roll surface.
- The average value of surface microhardness of the straightening roll having the chromium layer in accordance with Vickers was measured with the ZWICK 3212 hardness tester, and amounted to 915 HV. The average hardness after surface induction hardening of the layer, i.e. beneath the chromium layer of the straightening roll amounted to 60 HRC, which corresponded to a hardness of 790 HV. The hardness measured for the induction-hardened layer confirmed that hardening was adequate. The hardness measured in the tempered core amounted to 265 HV, which confirmed that its hardness, i.e., strength, was higher than that in the soft state, which confirmed the efficiency of thermal treatment in the core. For an additional increase in straightening-roll strength, a lower tempering temperature after quenching should be chosen.
From the macrographs of the straightening-roll surface it can be inferred that there are cracks across the entire straightening-roll surface. The size of the web-like cracks can be defined as the area covered by the web-like cracks, which ranges between 0.1 mm$^2$ and 0.7 mm$^2$. The majority of the web-like cracks is in the range of the axle ratio defining the oblongness of the crack. The ratio of dimensions of mutually rectangular axes of the web-like cracks ranges between 1.0 and 1.3, which means that the cracks are longish. The crack dimensions are by 30% longer with reference to the roll axis.

With statistical assessment of the measurements of the thickness of the hardened surface layer it was found that the average thickness of the surface-hardened layer was 1.065 mm and the average thickness of the chromium layer was 0.24 mm.

The additional macro and micrographic inspections confirmed that the hard chromium coating was porous, the fraction of porosity being 5.5%. It is very important that the pores were not concentrated; therefore, they did not represent a major risk of failure or descaling of the chromium coating. The failure and descaling of the chromium coating can be attributed to the procedure of chromium coating; therefore a change of parameters was recommended.

The fluorescent magnetic particle inspection confirmed that there were the web-like cracks already identified with the macroscopic treatment of the replicas produced at the straightening roll.

4. References
