



ANALYSIS OF PAINT LAYERS

Introduction

A painted steel nut, reference "Dwell MSV", which had been removed from steelwork, was supplied by MBC Building Contractors NW Ltd for examination.

Information provided indicates that the coating layers may include intumescent paint.

Sample Preparation

A section through the nut was taken by sawing and was then mounted in cold setting resin. The sample was prepared by wet grinding with silicon carbide papers followed by polishing with diamond. The sample was sputter coated with gold to render the surface conductive.

Scanning Electron Microscopy

A Leo 435VP scanning electron microscope fitted with an Oxford Instruments Isis x-ray energy dispersive analyser (EDS) was used to examine the sample. The analytical system will detect elements with atomic number greater than four.

Back-scattered electron images, which show sensitivity to average atomic number, were produced. In these images, low average atomic number appears darker than high average atomic number. Image magnifications quoted are nominal screen magnifications but images of a calibration grid are included, figures 15 & 16.

A back-scattered electron image of the section, figure 1, indicates seven coating layers, "1" to "7", are present on the nut. An image from a different region, figure 2, again shows seven coating layers. Whilst most of the layers appear to be reasonably uniform, layer "4" shows large variations in thickness. Layers "1" to "4" and "4" to "7" are shown in more detail in figures 3 and 4, respectively.

An x-ray spectrum from the nut substrate, figure 5, shows the main elements detected are iron, carbon, oxygen and silicon, indicating steel or wrought iron. Numerous large inclusions are visible in the nut substrate, see figure 4, suggesting the nut material is probably wrought iron.

An x-ray spectrum from coating layer "1", figure 6, shows the principal elements detected are oxygen, iron and carbon, indicating iron oxide, which probably formed during high temperature processing of the nut.

An x-ray spectrum from layer "2", figure 7, shows the main elements detected are carbon, oxygen, calcium, zinc, silicon, phosphorus, titanium, magnesium, iron, chlorine and aluminium. Further spectra show that the layer contains particles including zinc phosphate, figure 8, magnesium silicate (talc), calcium carbonate (limestone) and titanium dioxide white pigment in a partially chlorinated organic matrix. Zinc phosphate is commonly used to confer corrosion resistance in primers for steel.

An x-ray spectrum from layer "3", figure 9, shows the principal elements detected are calcium, oxygen, carbon, silicon, magnesium, phosphorus, zinc, iron, chlorine, aluminium and titanium. Additional spectra show the layer contains substances including calcium carbonate, magnesium silicate, aluminosilicate and zinc phosphate in a partially chlorinated organic matrix.

An x-ray spectrum from layer "4", figure 10, shows the main elements detected are carbon, oxygen, chlorine, titanium, phosphorus, aluminium, silicon, calcium and iron. An x-ray spectrum from the mid-grey phase in layer "4", figure 11, shows the principal elements detected are phosphorus, oxygen, carbon, nitrogen, chlorine, titanium and calcium, suggesting the phase is ammonium

phosphate, which is commonly used in intumescent paints. Further x-ray spectra show the layer also contains titanium dioxide white pigment and possibly iron phosphate in a chlorinated (halogenated) organic matrix. The combination of ammonium phosphate with a halogenated organic substance is commonly used in soft-char intumescent paints.

An x-ray spectrum from layer "5", figure 12, shows the main elements detected are oxygen, silicon, carbon, aluminium, titanium, magnesium, calcium, phosphorus, chlorine, potassium and iron. Additional x-ray spectra show the layer contains substances including calcium carbonate, potassium aluminosilicate, magnesium silicate, titanium dioxide and calcium phosphate in a partially chlorinated organic matrix. No evidence of ammonium phosphate was found.

An x-ray spectrum from layer "6", figure 13, shows the principal elements detected are oxygen, silicon, aluminium, calcium, titanium, carbon, phosphorus, potassium and chlorine. Further x-ray spectra show the layer contains compounds including potassium aluminosilicate, calcium carbonate and titanium dioxide in a partially chlorinated organic matrix. The location of the small amount of phosphorus detected has not been identified but no ammonium phosphate was found.

An x-ray spectrum from layer "7", figure 14, shows the main elements detected are oxygen, aluminium, silicon, carbon, titanium, calcium, magnesium and chlorine. Additional x-ray spectra show the layer contains substances including calcium carbonate, potassium aluminosilicate and titanium dioxide in a partially chlorinated organic matrix.

Reference to an image of a calibration grid, figure 15, allows the layer thicknesses in figures 1 & 2 to be measured. The overall thickness of the paint layers, "2" to "7", shown in figure 1 varies from about 957 μm to about 1616 μm , largely because the thickness of layer "4", the intumescent layer, varies from 300 μm to about 994 μm . The overall thickness of the paint layers, "2" to "7", shown in figure 2 varies and reaches a maximum of about 1533 μm , whilst the thickness of layer "4" varies from 407 μm to about 1001 μm .

Conclusions

1. The nut material is probably wrought iron rather than steel.
2. Seven coating layers are present on the nut.
3. Layer "1", the inner layer, is iron oxide, which probably formed during high temperature processing of the nut.
4. Layers "2" to "7" are paint layers.
5. Layer "2" has a partially chlorinated organic matrix, which includes:
 - zinc phosphate corrosion inhibitor,
 - magnesium silicate (talc),
 - calcium carbonate (limestone),
 - titanium dioxide white pigment.
6. Layer "3" has a partially chlorinated organic matrix, which includes:
 - calcium carbonate,
 - magnesium silicate,
 - aluminosilicate,
 - zinc phosphate.
7. Layer "4" has a chlorinated (halogenated) organic matrix, which includes:
 - ammonium phosphate,
 - titanium dioxide white pigment,
 - possibly iron phosphate.
8. Layer "5" has a partially chlorinated organic matrix, which includes:
 - calcium carbonate,
 - potassium aluminosilicate,
 - magnesium silicate,
 - titanium dioxide,
 - calcium phosphate.
9. Layer "6" has a partially chlorinated organic matrix, which includes:
 - potassium aluminosilicate,
 - calcium carbonate,
 - titanium dioxide.
10. Layer "7" has a partially chlorinated organic matrix, which includes:
 - calcium carbonate,
 - potassium aluminosilicate,
 - titanium dioxide.
11. Layer "4" contains ammonium phosphate together with a halogenated organic substance, which is a combination commonly used in soft-char intumescent paints.
12. In the regions examined, the thickness of layer "4" varies from 300µm to about 1001µm.
13. The overall thickness of the paint layers, "2" to "7", varies from 957µm to about 1616 µm, largely because of the variation in thickness of layer "4".

Andrew Cole
12th May 2023

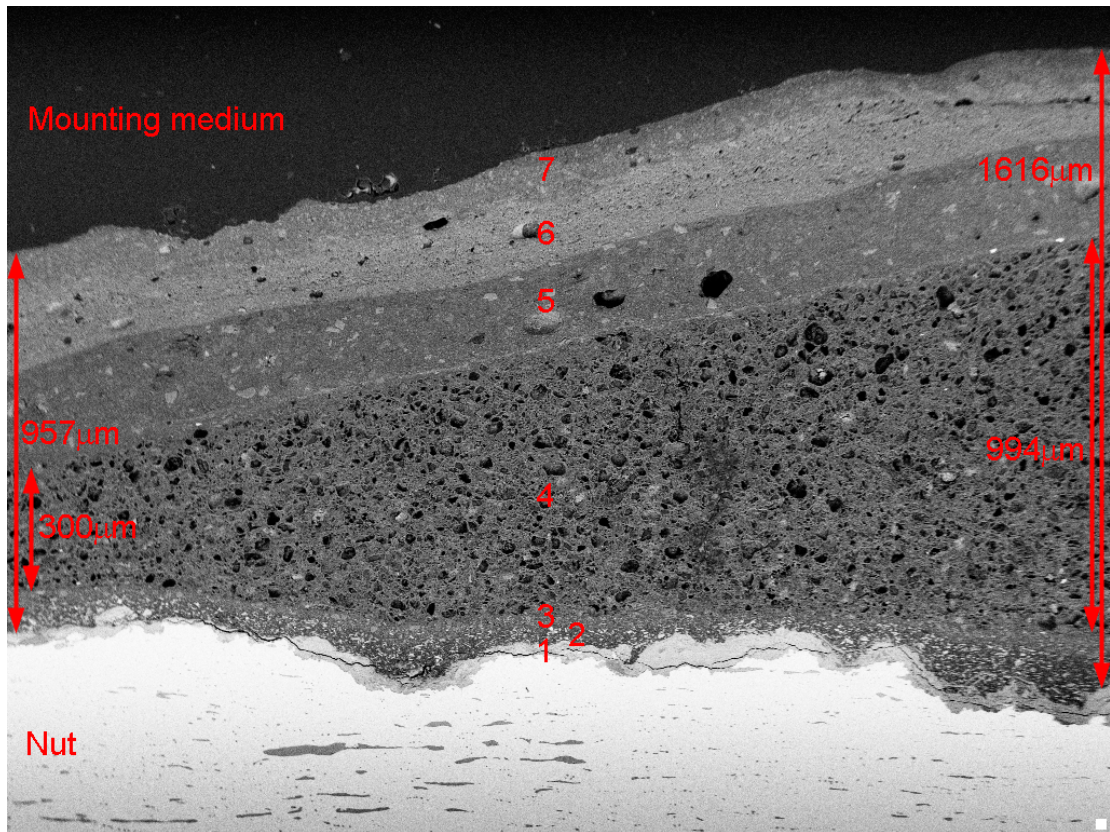


Figure 1. Back-scattered electron image of section through coating layers on nut. x100.

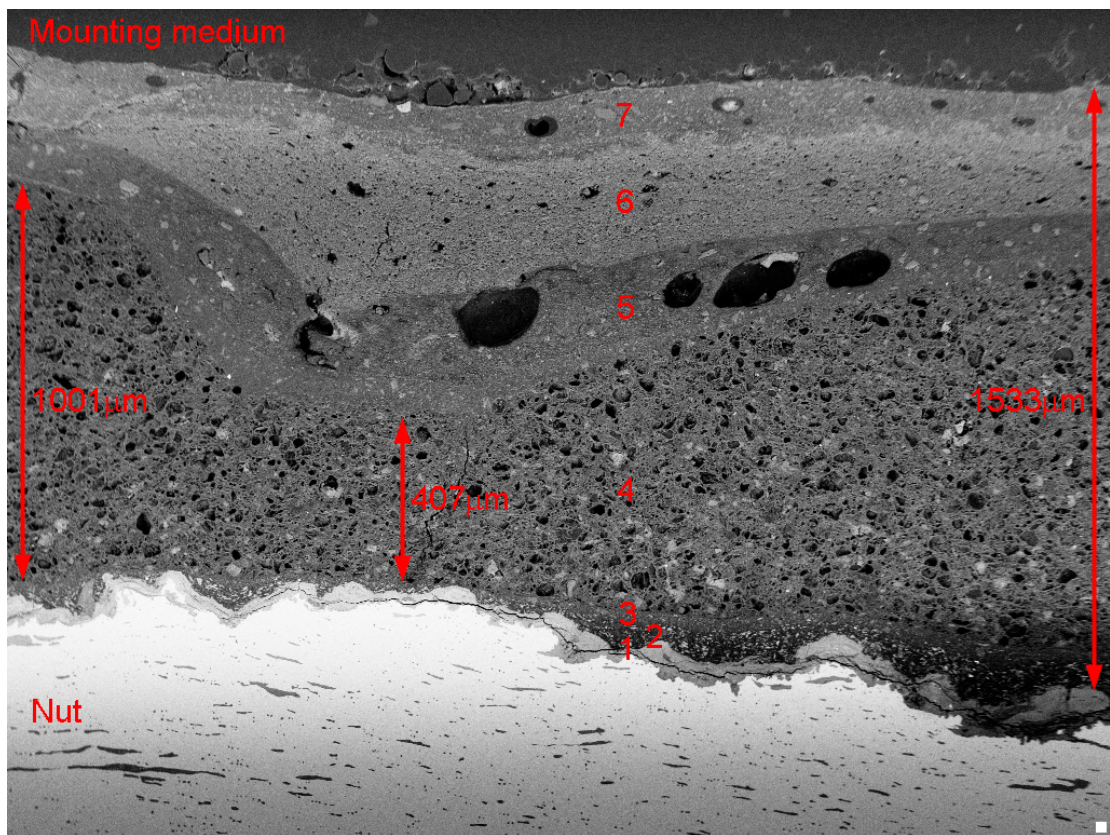
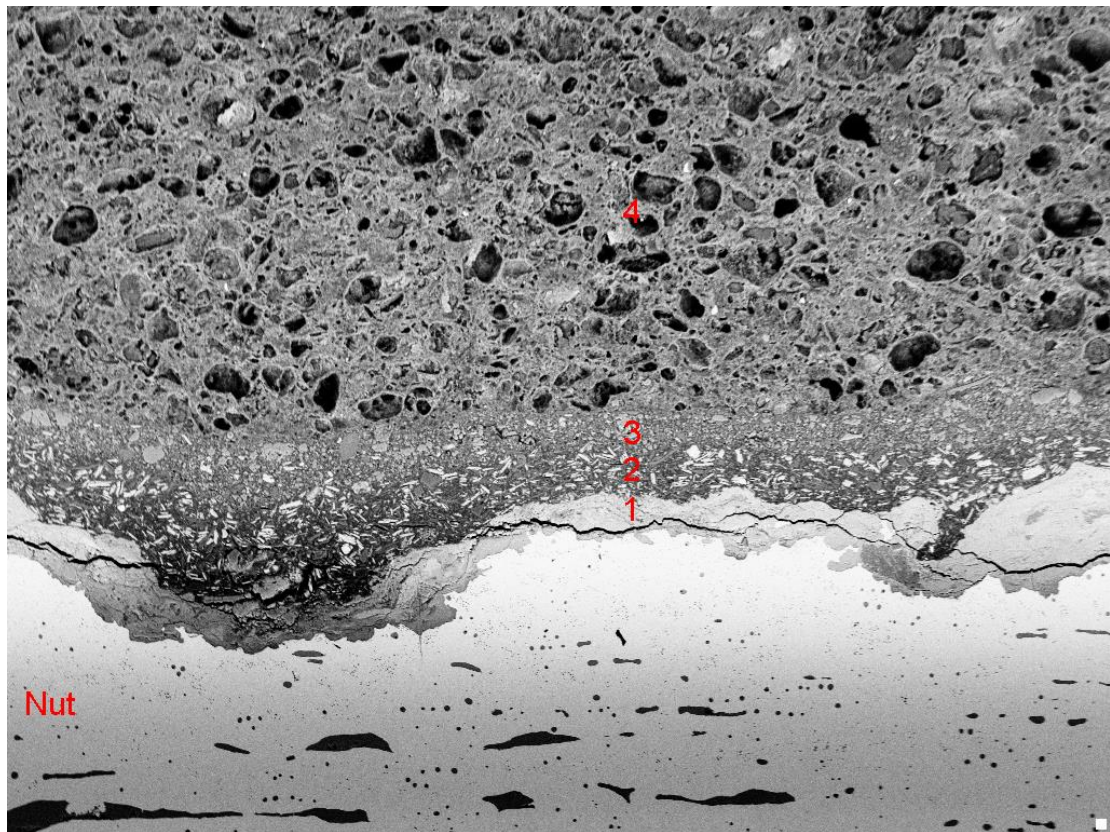
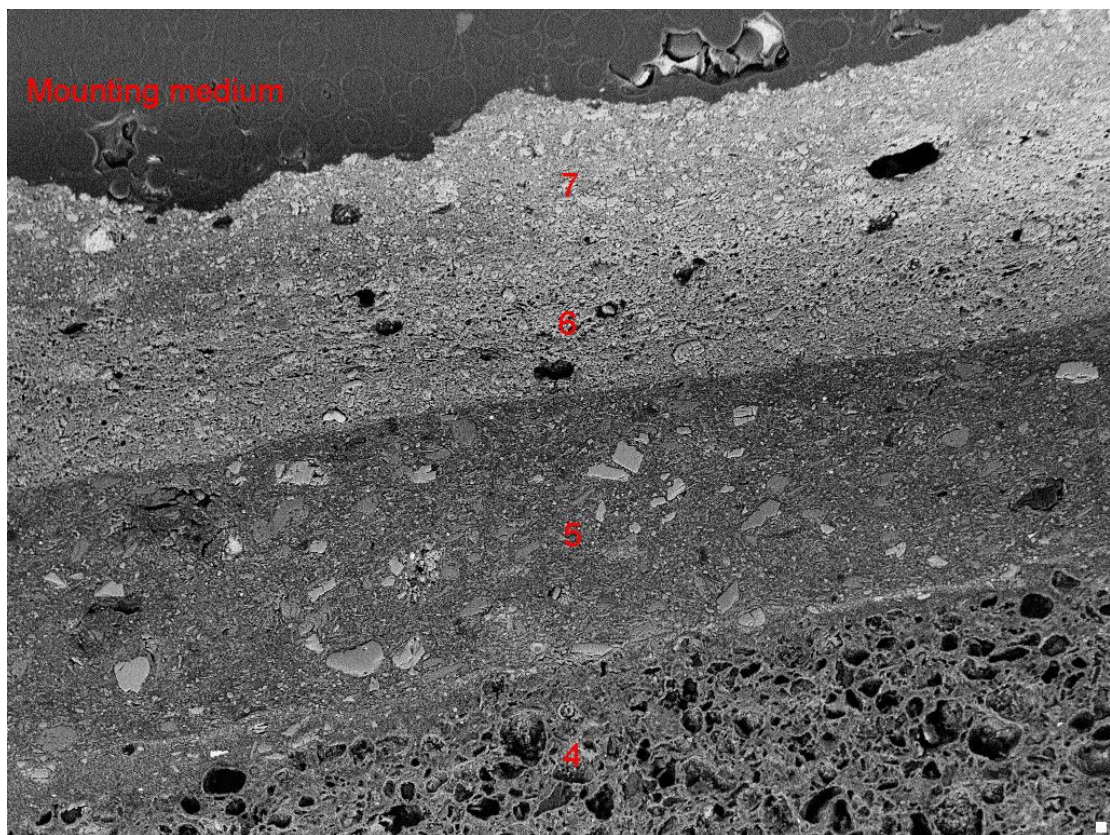


Figure 2. Back-scattered electron image of section through coating layers on nut. x100.



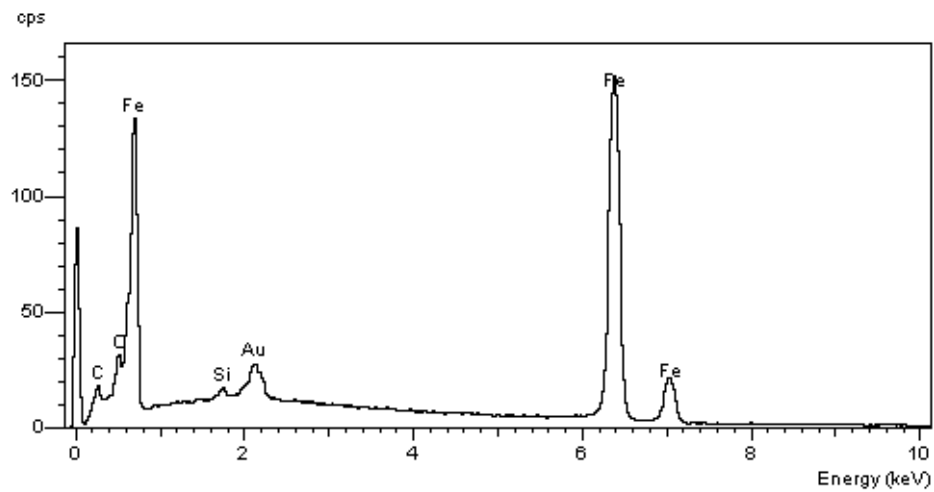
8338

Figure 3. Back-scattered electron image of section through coating layers "1" to "4". x300.



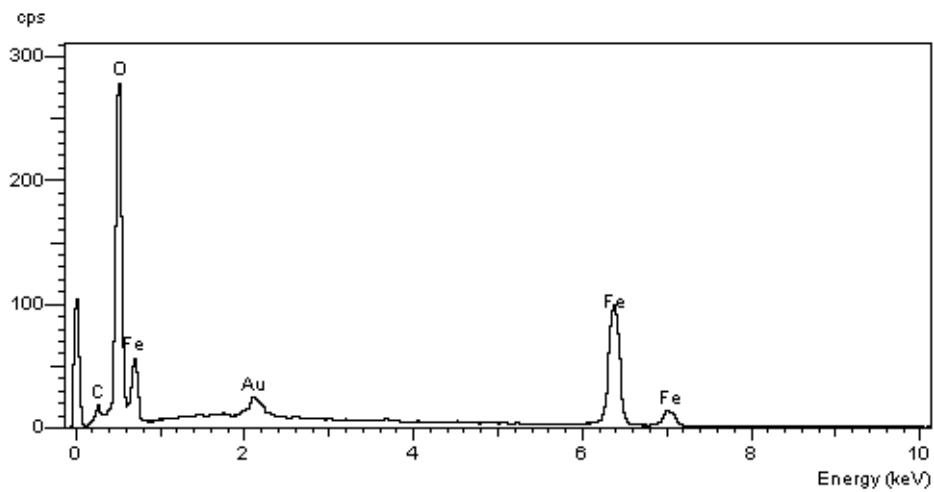
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Figure 4. Back-scattered electron image of section through coating layers "4" to "7". x300.



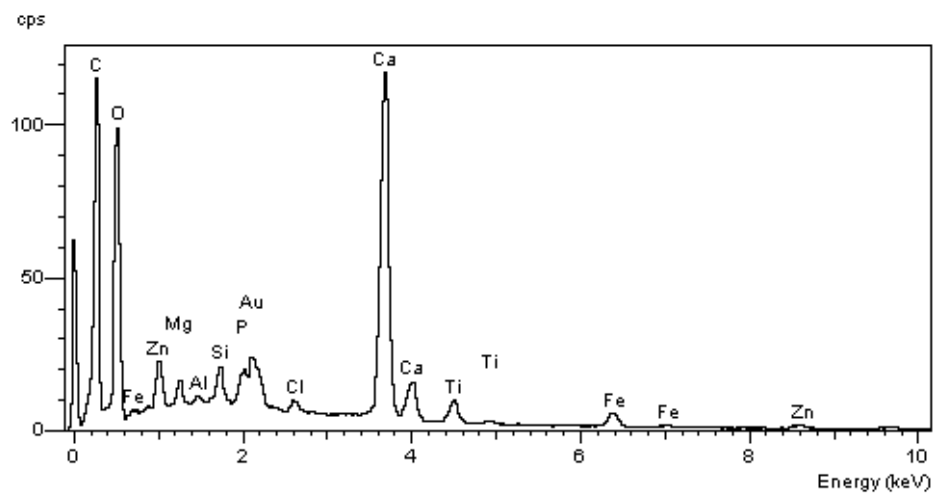
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Figure 5. X-ray spectrum of nut substrate.



j4258b

Figure 6. X-ray spectrum of layer "1".



j4258c

Figure 7. X-ray spectrum of layer "2".

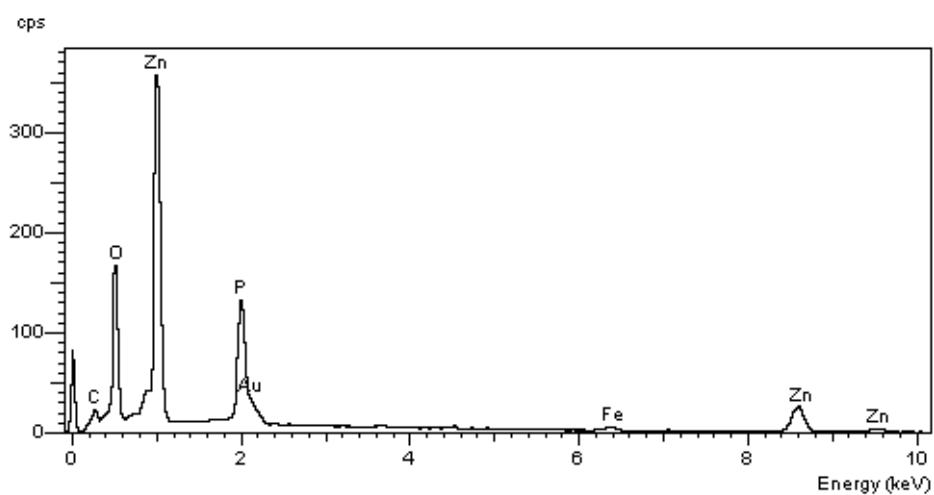


Figure 8. X-ray spectrum of light-grey particle in layer "2".

j4258d

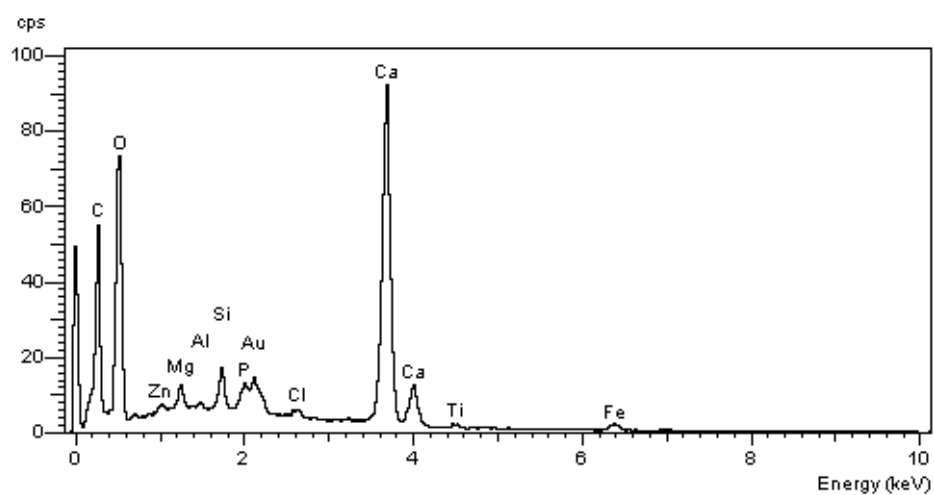


Figure 9. X-ray spectrum of layer "3".

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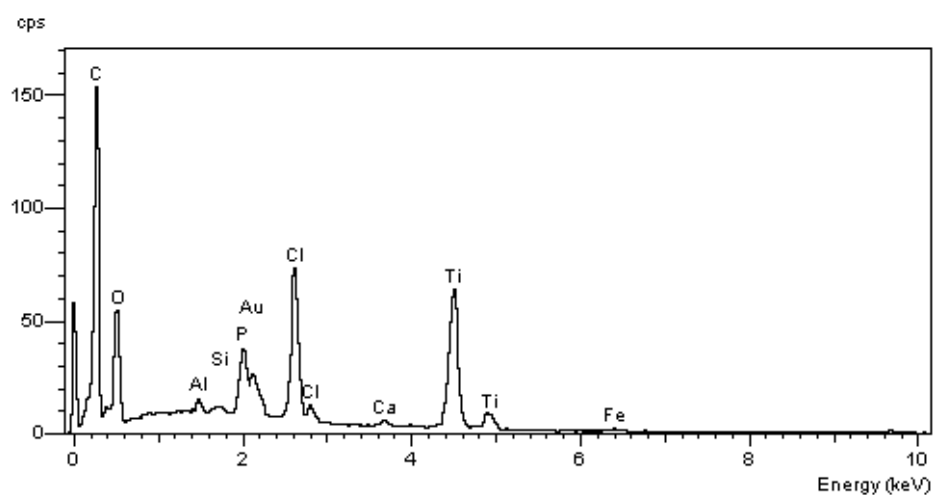
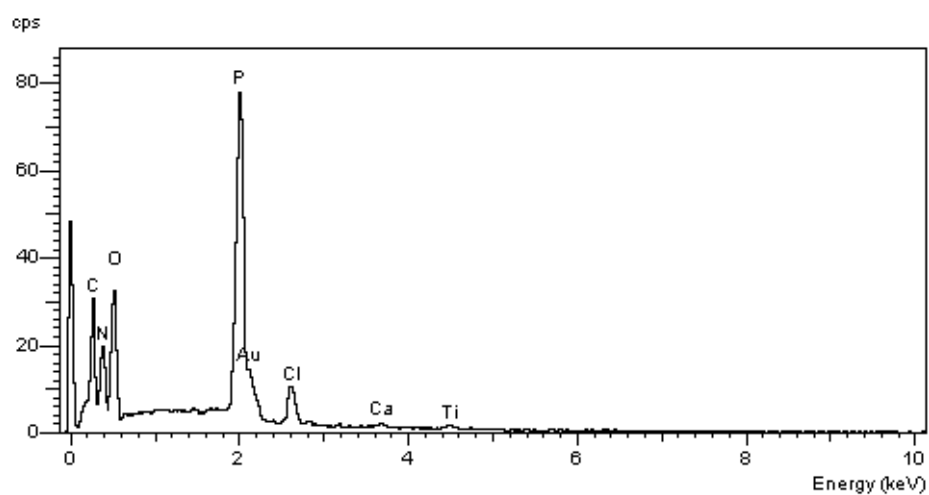


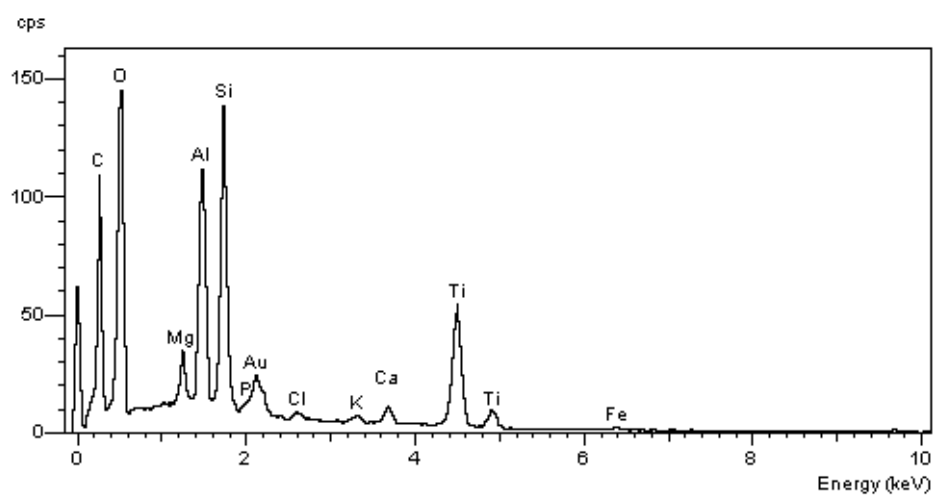
Figure 10. X-ray spectrum of layer "4".

j4258f



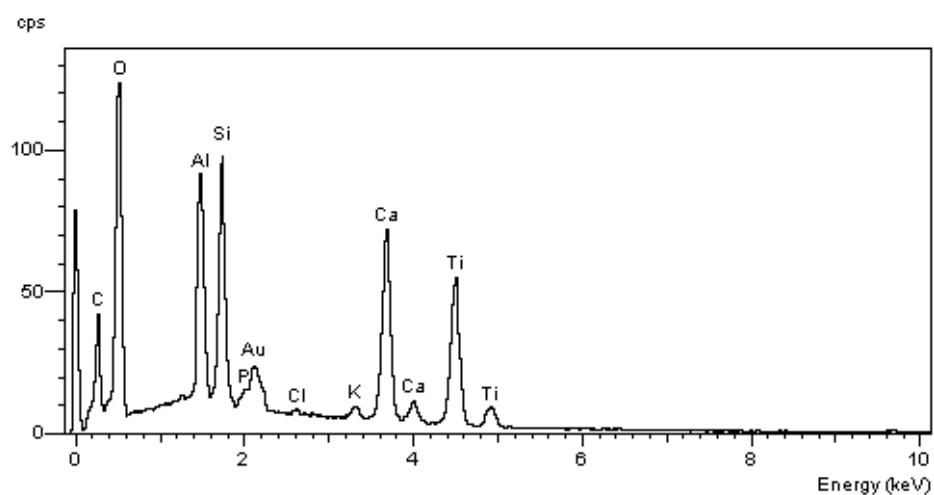
j4258g

Figure 11. X-ray spectrum of mid-grey phase in layer "4".



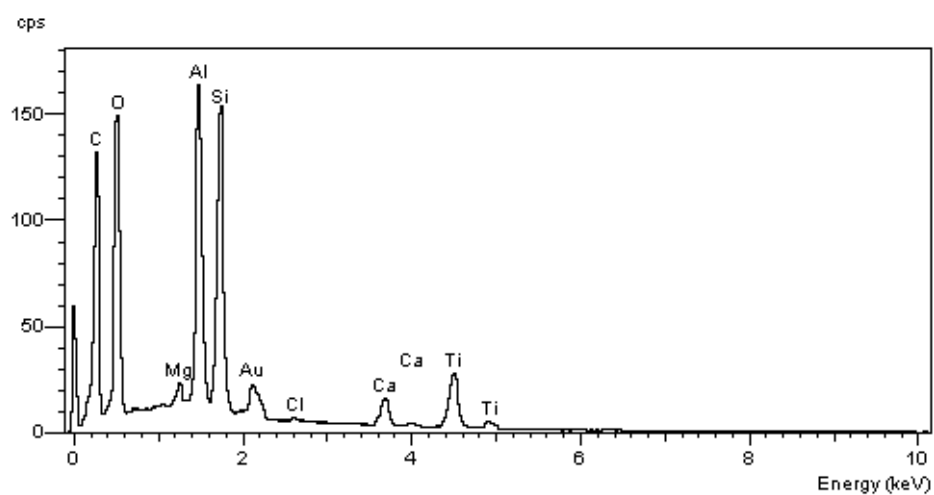
j4258h

Figure 12. X-ray spectrum of layer "5".



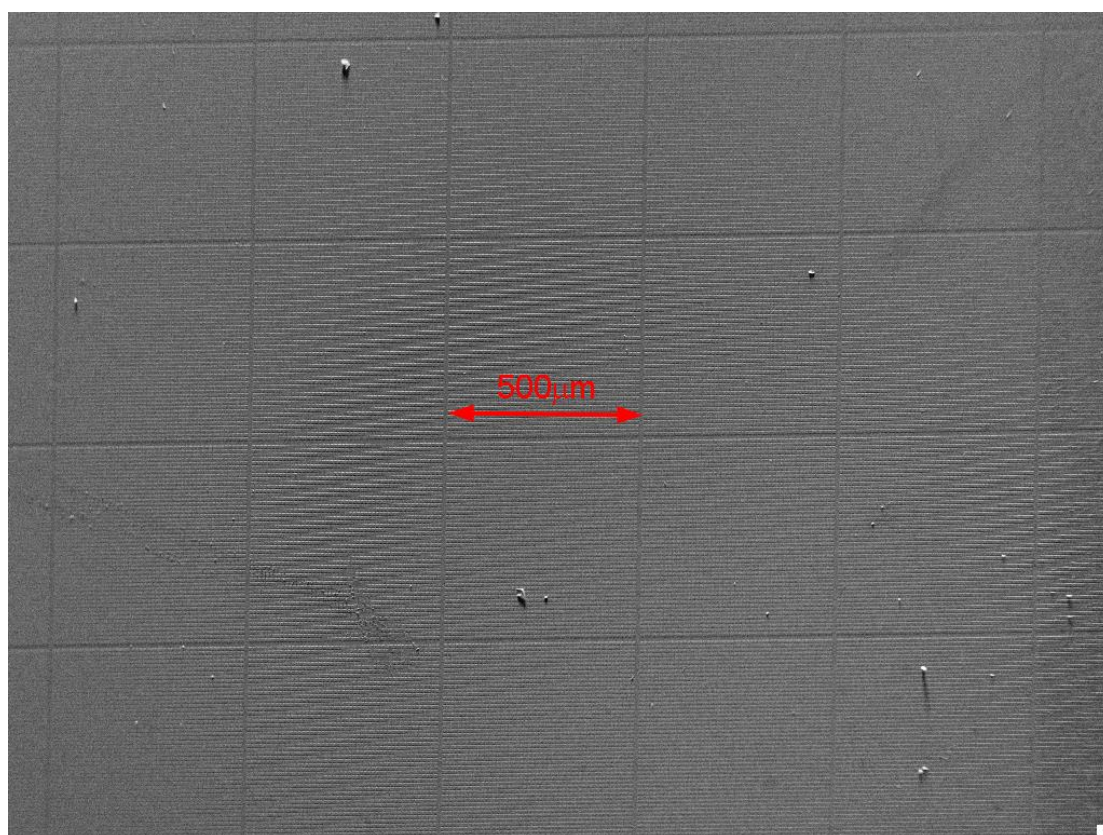
j4258i

Figure 13. X-ray spectrum of layer "6".



j4258j

Figure 14. X-ray spectrum of layer "7".



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Figure 15. Secondary electron image of calibration grid. x100.

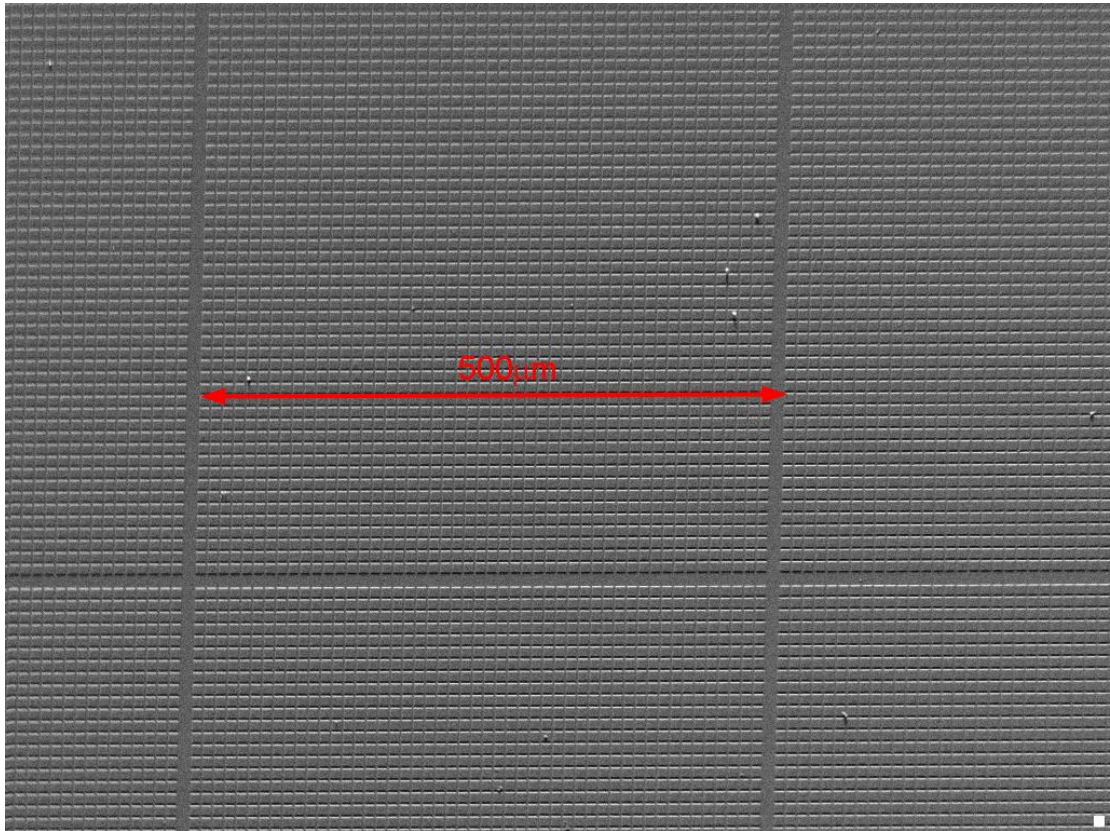


Figure 16. Secondary electron image of calibration grid. x300.