

Ion Beam Etching **Applications** **in Material Characterization**

**Parameter Studies using the PECS Ion Beam
Etching and Coating System**

Final Report

Parameter Studies with the Ion Beam Etching and Coating System PECS

Isolde Gräf, Fachgebiet Strukturforschung, Technische Universität Darmstadt in
cooperation with Gatan GmbH, Munich

List of Contents

	Page
1. <u>INTRODUCTION</u>	1
2. <u>TEST PROCEDURE</u>	1
2.1 ION BEAM ETCHING WITH PECS	2
2.1.1 Acceleration Voltage and Ion Current	2
2.1.2 Ion Incidence Angle (specimen tilt)	5
2.1.3 Etching of Embedded Specimens	5
2.1.4 Metallographic Specimen Preparation	6
3. <u>TEST RESULTS</u>	7
3.1 ETCHING PARAMETERS FOR VARIOUS MATERIALS	8
3.1.1 <u>Steel and Iron Alloys</u>	9
3.1.1.1 PM Steel Supermet Fe64 (Figures 7 and 8)	9
3.1.1.2 Tool Steel 100Cr6 (Figures 9 and 10)	9
3.1.1.3 Ferrite Stainless Steel X7Cr13 (Figures 11 and 12)	11
3.1.1.4 Alloy FeCo (Figures 13 to 15)	11
3.1.1.5 Hardened Steel Ck15	14
3.1.1.6 Construction Steel Ck45 (ferritic/perlitic) (Figures 16 to 18)	14
3.1.1.7 Austenitic Steel 1.4301	15
3.1.2 <u>Titanium and Titanium Alloys</u>	17
3.1.2.1 High Purity Titanium (Figures 19 to 21)	17
3.1.2.2 Ti- Alloy with Si (Timetal 1100) (Figures 22 to 26)	19
3.1.2.3 Alloy Ti6V4	22
3.1.3 <u>Cu and Cu Alloys</u>	22
3.1.3.1 Cu Pole Pieces (high purity Cu, deformed and annealed with a tin coating ($< 2 \mu\text{m}$) (Figures 27 to 30)	22
3.1.3.2 Electrolyte Cu	24
3.1.3.3 CuCrZr Alloy, Material 2.1293	24
3.1.3.4 Machining Brass: CuZn39Pb3 (Figures 31 and 32)	25
3.1.3.5 Composite Wire CuNiAg	25
3.1.4 <u>Al and Al Alloys</u>	26
3.1.4.1 High Purity Aluminium (Figures 33 to 38)	26
3.1.4.2 Machining Alloy: AlMgSiPb / F28 (Figure 39)	30
	Page

3.1.4.3	Casting Alloy AlMg3 (non-hardenable)	31
3.1.4.4	Casting Alloy AlMg4.5Mn (non-hardenable) (Figures 40 and 41)	31
3.1.4.5	AlMg4.5Mn/15G, annealed / rolled, strained texture (non-hardenable) (Figures 42 to 44)	33
3.1.4.6	AlMg4.5Mn/25G, annealed / rolled, strained texture (non-hardenable)	35
3.1.4.7	Various Alloys	35
3.1.5	<u>Mg and Mg Alloys</u>	36
3.1.5.1	High Purity Mg	36
3.1.5.2	Mg Casting Alloy AM50 (Figures 45 to 49)	36
3.1.5.3	Mg Die Casting AM50	36
3.1.5.4	Mg Alloy AZ91 (Figures 50 to 52)	36
3.1.6	<u>Superconductors</u>	41
3.1.6.1	Superconductor Cu+NbTi	41
3.1.6.2	Superconductor CuSn+NbTi+Ta, covered with Cu Coating (Figures 53 to 57)	41
3.1.6.3	Superconductor Extruded Cu+Sn/NbTi+Ta with Cu Coating (very fine)	44
3.1.6.4	Superconductor AgMg Mantle, Ceramic Core of PbBeSr/CaCuO	44
3.1.6.5	Superconductor Ag Mantle / Ceramic Core (Composition unknown) (Figures 58 to 62)	44
3.1.7	<u>Permanent Magnets</u> , Composition: FeNdB(1%), hard-magnetic (Figures 63 to 65)	47
3.1.8	<u>Layered Systems</u>	49
3.1.8.1	Build-up Welds (Ni alloy on boilerplate HII) (Figures 66 and 67)	49
3.1.8.2	Build-up Welds (Ni alloy on boilerplate HII), Triple Layer, Rolled	49
3.1.8.3	Build-up Welds (Ni alloy on GGG40)	49
3.1.8.4	Welds (Hastalloy) with Ni Intermediate Layer on Plain Steel (Figures 68 to 72)	51
3.1.8.5	PM Steel with Antiabrasion Layer (austenitic)	54
3.1.8.6	Nickel Solder: Nimonic / NB30 (16h/710°C) (coarse phase) (Figures 73 to 75)	54
3.1.8.7	Nickel Solder: Nimonic / NB125 (fine phase) (Figures 76 to 80)	54
3.1.8.8	Nickel Solder: Steel HFX / NB30 (coarse phase)	54
3.1.9	<u>Puddled Steel</u> : Ferrite with Slag and Rust (Figures 81 to 84)	59
3.1.10	<u>Ceramics and Other Non-Metals</u>	61
3.1.10.1	Aluminum Nitride AlN	61
3.1.10.2	PIC 151 [Pb(Zr _{0.52} Ti _{0.48})O ₃] + 1-2% Ni, Sb (yellow)	61
3.1.10.3	PIC 141 [(Pb(Zr _{0.52} Ti _{0.48})O ₃) + ca. 1% Fe (black)	62
3.1.10.4	Zircon Oxide ZrO ₂	62
3.1.10.5	Al ₂ O ₃ -Standard	62
4.	<u>CONCLUDING REMARKS</u>	63
5.	<u>LITERATURE</u>	63
6.	<u>ACKNOWLEDGEMENT</u>	63

Parameter Studies with the PECS Ion Beam Etching and Coating System

Isolde Gräf, Fachgebiet Strukturforschung, Technische Universität Darmstadt

1. Introduction

Ion beam etching suggests itself for the preparation of materials for various investigations such as the thinning of samples for transmission electron microscopy or for structuring surfaces in the semi conductor industry. The method is based on a physical process in which the specimen to be examined is bombarded with high-energy particles (ions or neutral particles). Thereby collision cascades in the surface are generated leading to the discharge of near-surface atoms. The local material loss and the resulting morphology depend on the nature of the specimen, the crystal orientation and the defect structures (e.g. grain boundaries or dislocation glide bands). They lead to a texture and a contrast of the specimen's fabric in polished sections of the sample. Ion beam etching has proven to be a good supplementary method with reference to the conventional metallographic etching method. Frequently additional information can be obtained on the structure of materials. Sometimes the application of this 'modern' etching technique is the sole possibility of making statements regarding the metallographic sequences or processes, for example, for composite materials or material compounds. To date, ion etching has rarely been used as an alternative etching method. To counter-balance this deficit, comprehensive etching tests and parameter studies were performed on various materials. Homogeneous materials such as Cu or high purity Al were examined (for instance, E-Cu, R-Al) as were heterogeneous materials such as machining brass and weld connections.

2. Test Procedure

The etch tests were performed using the GATAN ion beam etching and coating system PECS. With the broad beam ion source in this system, etch marks of about 10 mm can be achieved at vertical incidence of the beam, dependent on the target material and the parameters. By tilting the specimen, i.e. by altering the incidence angle, the etch mark can be enlarged further if the specimen is rotated during the process. In this way, areas up to a diameter of about 20 mm can be etched. For optimum texture development, it is sometimes advantageous to vary the beam incident angle. For example, starting the etching process at a low angle (e.g. 45° to 55°) and then switching to a steep angle (e.g. 10° to 0°, i.e. vertical incident). For the microscopic examinations, usually the contrast must be further enhanced by using differential interference contrast (DIC).

The material loss is closely connected to the sputtering yield (S), i.e. ejected atoms per incoming ion (atoms / ion). It is dependent on ion-related characteristics such as ion mass, ion energy, ion current density and ion angle of incidence and on material characteristics such as atomic mass, crystal structure, crystal orientation, surface condition and surface xenolayers. According to Wechsung, for example, the sputtering yield amounts to 1.1 (atoms / ion) for Ti, 1.3 (atoms / ion) for Fe, 3.6 (atoms / ion) for Cu and 0.9 (atoms / ion) for Ta [vertical beam incident, 1 keV energy].

2.1. Ion Beam Etching with Pecs

2.1.1 Acceleration Voltage and Ion Current

The acceleration voltage for PECS ranges from 1.3 keV to 10 keV. The maximum ion current increases with increasing acceleration voltage. However, for metrological reasons, it cannot be adjusted above a voltage of about 7 keV to more than 614 μA . The ion current is controlled by the gas flow. The achieved diameter of the etch mark and the intensity of the etching in the center is in the first instance dependent on the selected acceleration voltage, assuming the same material, same etching time and vertical angle of incidence. The largest etch mark is generally achieved at 6 keV to 6.5 keV. With increasing voltage, the etch mark becomes somewhat smaller, the etching intensity in the center meanwhile stronger. Similarly, with decreasing voltage, the etch mark becomes slightly smaller, however, with simultaneous weakening of the etching intensity.

The selected ion current similarly has an impact on the etch effect. In order to keep the ion beam stable during the etching process, the current should be if possible 5 to 25 % below the obtainable maximum, which is achieved by increasing the gas flow. At an acceleration voltage of 6 keV, the maximal reachable ion current under normal circumstances is between 540 μA and 560 μA . Accordingly at 6 keV, an ion current of between 400 μA and 530 μA should be set during the etching process. Depending on the state of the etching chamber or the ion source, i.e. after a longer period of use or freshly cleaned, sometimes the above-mentioned maximum is not reached or not displayed, so that a lower ion current has to be selected. The etching effect can thereby be somewhat reduced.



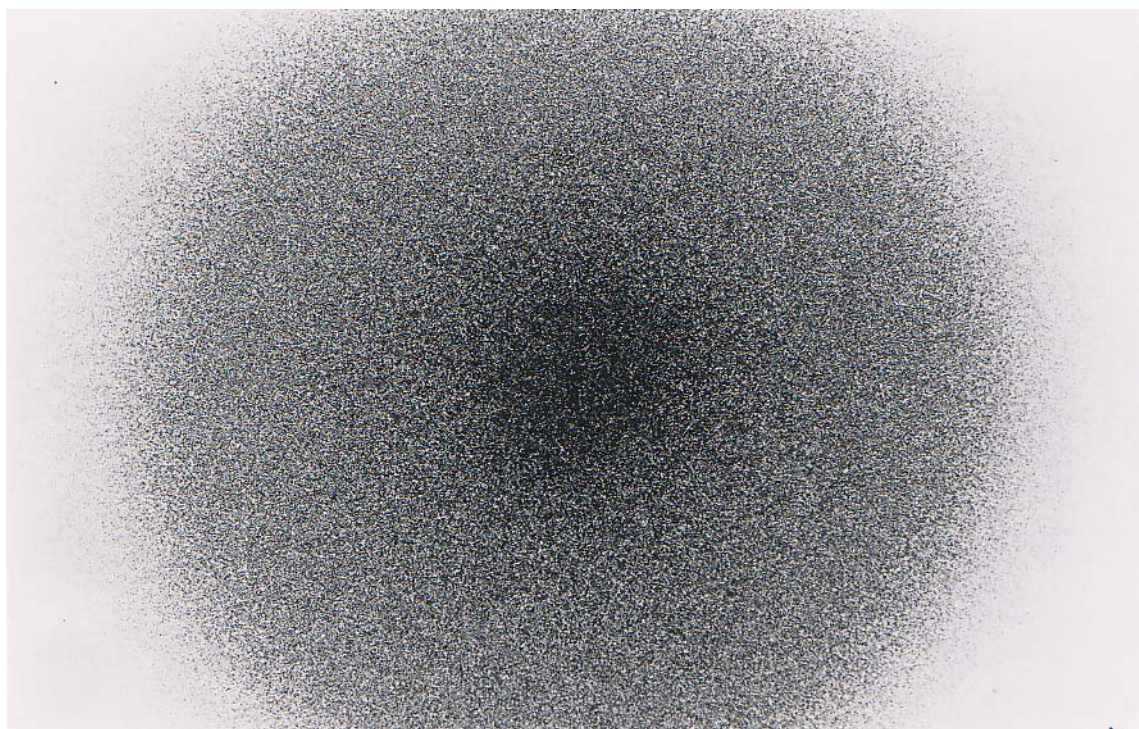
12.5x

Figure 1: PM Steel Fe64 (Specimen 4) ion beam etched, Parameter: 6.5 kV / 450 μA / 8 mins



12.5x

Figure 2: PM Steel Fe64 (Specimen 5) ion beam etched, Parameter: 7 kV / 460 μ A / 8 mins



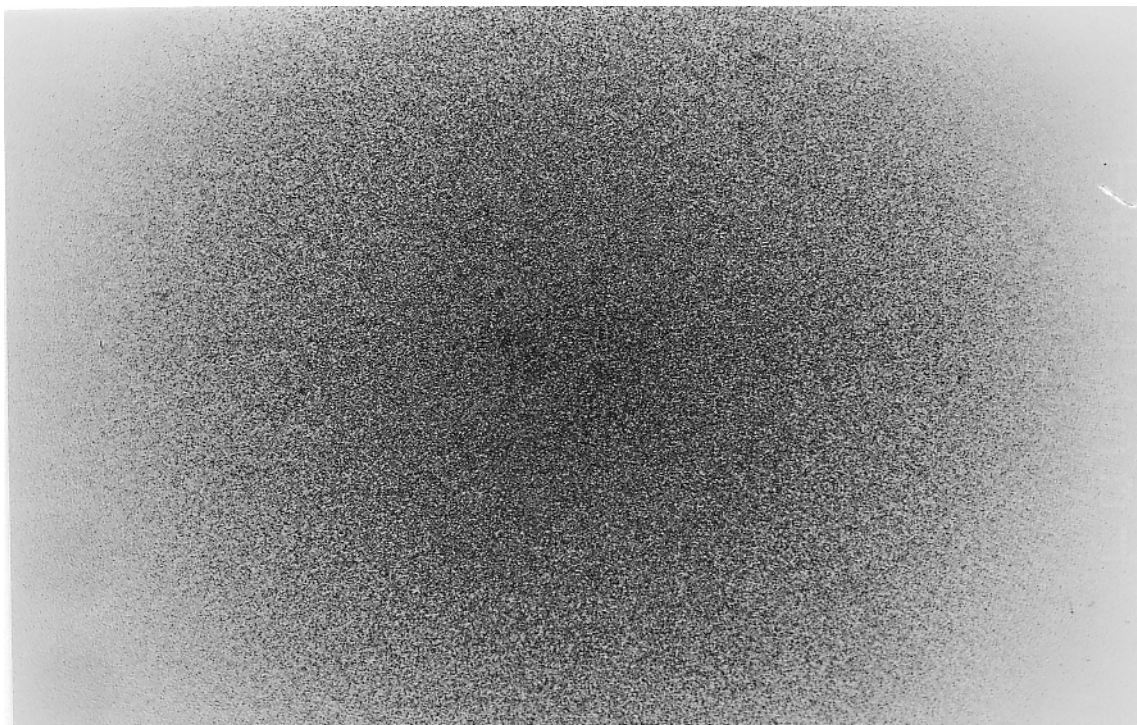
12.5x

Figure 3: PM Steel Fe64 (Specimen 1) ion beam etched, Parameter: 8 kV / 456 μ A / 8 mins



12.5x

Figure 4: PM Steel Fe64 (Specimen 6) ion beam etched, Parameter: 6 kV / 415 μ A / 8 mins



12.5x

Figure 5: PM Steel Fe64 (Specimen 8) ion beam etched, Parameter: 6kV / 270 μ A / 8 mins

A number of different parameter settings were tried for polished specimens made of high-carbide content PM Steel Supermet Fe64 (Krupp PM designation, composition as for X225CrVMo134) using powder-metallurgical methods. Figures 1-3 show the impact of the acceleration voltage for a constant ion current of about 455 μA . Figures 4 and 5, by contrast, show the impact of different ion currents at a constant voltage of 6 keV.

2.1.2 Ion Incidence Angle (specimen tilt)

The ion incidence angle can be adjusted between 0° (vertical bombardment) and 90° (beam parallel to the specimen surface). A specimen tilt readout is located next to the airlock, at the right hand side of the etch chamber. The specimen can either be fixed at an angle and etched in this position or additionally, a rotating and/or oscillating movement can be set. The incident angle has a decisive influence on the development of textural or structural effects. By means of tests performed on the steel Ck45, the following can be ascertained using light-microscopic observation methods: the lower the angle, i.e. the steeper the incident, the more intensely the grain boundaries are etched. The grain boundaries are normally only moderately exhibited at bombardment angles between 50° and 60° . Between 15° and 30° , however, they are etched considerably more. They appear in this process to be more slanted due to the discharge corresponding to this incidence angle.

In general, the contrast must - as was described above - be enhanced for light microscopy by using differential interference contrast (DIC). For vertical ion bombardment, the grain boundaries are so strongly exhibited that the samples to a very large extent are also suitable for normal reflected light micrographs with magnifications up to 200 times. Besides vertical bombardment, an etching angle of 45° appears to provide optimum texture development. Some materials - such as high purity aluminium - can apparently only be properly etched at less than about 45° . The just described etching effects and their dependence on the ion angle of incidence are only relevant for a specimen height of about 10mm. For higher specimens, a steeper angle should be selected to achieve the same etch effect, e.g., for a height of about 12.5mm, an angle of about 30° should be used.

Particularly pronounced is the impact of the bombardment angle on the texturing of aluminium alloys. Experience has shown that a grain surface etching is generated using flat incidence angles between 65° and 55° , whereby the grains, depending on the orientation (possibly also depending on precipitates) appear rough to grainy. The grain boundaries are only finely etched. At about 45° the grain boundaries are more strongly etched and the grain surfaces tend to be smooth. Using smaller or steeper bombardment angles of about 30° , dot-like artifacts can occur on the relatively smooth grain surfaces and, when using even smaller angles ($< 15^\circ$), additionally on the grain boundaries. When longer etch times are involved, often 'string of pearls'-like structures appear. To what extent this is to be linked to precipitates has yet to be clarified.

2.1.3 Etching Embedded Specimens

The specimens to be etched should not be embedded to ensure solid heat and electrical contact to the specimen holder. If embedding cannot be avoided due to the smallness of the specimen or a complicated specimen shape, an embedding agent should be used which does not outgas nor melt, i.e. is, if possible, heat-resistant. Otherwise the vacuum of $<5 \times 10^{-5}$ torr required for the etching process cannot be reached. A track of silver dag should be laid for the

embedded specimens to generate the contact with the mounting. Tests demonstrated that it is advantageous if embedded specimens are covered with a pinhole diaphragm. Such a diaphragm can be made from somewhat thicker aluminium foil and cut a hole in it to match the form of the material to be etched. The diaphragm, which may not be larger than the external diameter of the specimen mounting, can be fastened at the edge of the fixture using kneadable carbon dag. Alternatively the embedding medium can be completely covered with silver dag. If embedded specimens are not protected in this way, only a tiny etch mark on the target to be etched is generated as a result of extreme charging. Occasionally embedded specimens (particularly Al alloys) cannot be etched despite the afore-mentioned measures. The only thing to do then is to extract the specimen.

In this connection, here is a practical tip: specimens with a small diameter can be quasi clamped into the specimen fixture with aluminium foil. To do so several rings are made by rolling a piece of aluminium foil into rings, which are then placed around the specimen and pressed firm.

2.1.4 Metallographic Specimen Preparation

Decisive for the success of ion beam etching is the extremely careful specimen preparation. The specimens should be scratch-free und polished thoroughly. Scratches are usually extended laterally when subjected to ion beam etching and existing deformation layers reinforced so that the actual texture structure is conceivably falsified. Under certain circumstances, a repeat of the fine and final polishing process cannot be avoided after an initial etching in order to eliminate the deformation layers completely. If at all possible, the final polishing should be checked light-microscopically with differential interference contrast (DIC). Dried up damp spots and residual polish must similarly be removed carefully from the polished surface as both cannot be removed by the etching process but rather can lead to artifacts.

In addition, resulting from a longer period of storage of an already prepared specimen, oxide layers can form on the polished surface, which lead to an impairment of the texture development. In other words, the state of the specimen surface can, on the one hand, impact on the size of the etch mark and, on the other hand, on the etching intensity. Whereas, in a first etching test at 5 keV, 270 μA and a timespan of 8 mins, the diameter of the etch mark is estimated at about 5 mm, in a second test, using the same parameters and the now freshly polished specimen, a diameter of about 9 mm was achieved. The intensity distribution of the etching had similarly altered. Figure 6 shows the etch mark for the PM Steel Supermet F64 after the second test.

The polished specimens can be prepared for ion etching using commercially available metallographic abrasion and polishing agents. The suspension agents OP-S or OP-U, together with the OP-Chem polishing cloth, should be used for the final polish. In order to remove deformation layers more easily, a suspension made of liquid red polishing agent and 25 % hydrogen peroxide (H_2O_2) can be added during the final polishing process. Excepted from this are materials susceptible to corrosion.

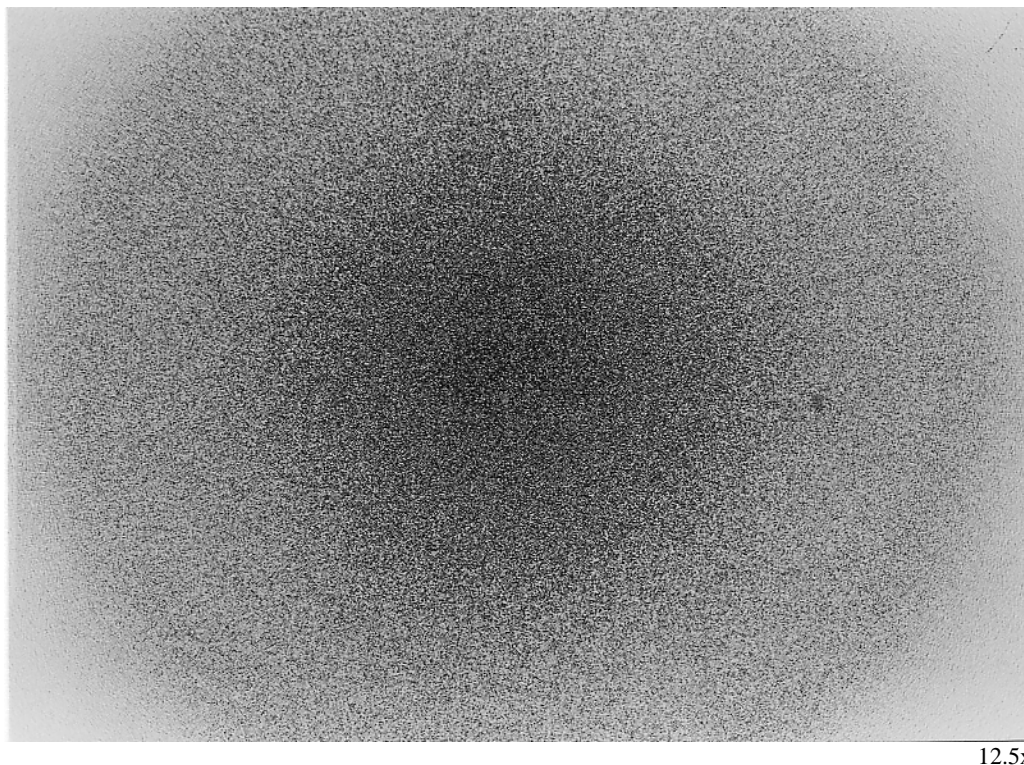


Figure 6: PM Steel Fe64 (Specimen 7) ion beam etched, Parameter: 5 kV / 270 μ A / 8 mins

3. Test Results

Basically, a satisfactory texture development can be achieved by ion beam etching for all metallic materials used in the Parameter Studies. Texturing of ceramic materials turned out to be more difficult. Here only for 3 out of 7 of the differing ceramics examined could a satisfactory grain boundary etching be achieved. Presumably the linkage forces between the grains are so high that, due to the intense heat generated, the results lead to indefinable artifacts rather than a development of the grain boundaries. Possibly further parameter studies will have to be performed for the ceramic materials, conceivably using reactive ion beam etching (RIBE).

For ion beam etching of composite systems, amongst other things, the sputtering yield (S) plays an important role. Materials with similar sputtering yields can fundamentally be textured jointly without difficulty. Greater problems are caused, if the specimen consists of components with greater differences in S (e.g. Cu/Ti). In such cases, all composite partners can usually be imaged with considerable contrast by using differential interference contrast (DIC) or polarization microscopy. A further contrast enhancement can be achieved if, after etching with argon ions, a reactive ion beam etching with oxygen is supplementarily conducted. A brownish, occasionally reddish, tarnish layer is generated which is differently colored according to the grain orientation.

One problem arises that the perlite in steel or gray cast iron is not textured by ion beam etching in the way this usually occurs in metallography. Even the test using oxygen instead of argon for texture development failed. On the other hand, there was an exception among the experiments conducted. Perlite developed practically in the customary way in a polished Ck45 specimen. There must therefore be test conditions – possibly with an increased neutral

particles proportion – which permit to exhibit perlite in steel as usual. Perlite textures are etched in other materials – such as with the Mg alloy AZ91 – without any difficulty.

3.1 Etching Parameters for Various Materials

From the range of experiments performed, the following etch parameters are listed which provided good etching results for the different materials. They can serve as examples for new etching tests with different new materials. The commonest acceleration voltage is 6 keV, for materials with a greater sputtering yield, such as Cu or PM steel, 5 keV. In the case of some materials such as Ti and Ti alloys, for permanent magnets or occasionally also for Al alloys, the voltage was increased to 6.5 keV. An even higher acceleration voltage of 8 keV to 9.5 keV proved to be advantageous for etching ceramic materials. Similarly the contrasting with oxygen was performed using voltages between 8 keV and 9.5 keV. Lower voltages produced neither tarnish nor color effects.

The etching intensity, i.e. the etch attack on the texture, was controlled predominantly, besides the selected bombardment angle, via time. For copper with a high yield (S), even 2 mins to 3 mins suffice for optimum texture development. For titanium with a lower sputtering yield, up to 20 mins etching time were needed.

For overview reasons, the etching conditions are applied in a similar way to the listed test protocols, respectively without units. In addition, a number of special features are noted. The parameters are presented as follows:

Specimen Designation Voltage [keV] / Ion Current [μ A] / Time [min], Etching Angle, Rotation (if not mentioned, then fixed at an angle)

The etching process can be interrupted without difficulty at any time and supplemented by other tests. The respective supplementary process data are given a "+" and written one under the other. The results of the individual tests are commented on using keywords. The following have the meanings:

KG = Grain Boundary	DIC = Differential-Interference Contrast
KF = Grain Surface	POL = Polarization, Polarized Light
Mk = Mixed Crystal	

The enlargement or scale details are:

12.5x \equiv 1mm ↔	25x \equiv 0.5mm ↔	50x \equiv 250 μ m ↔	100x \equiv 100 μ m ↔
200x \equiv 50 μ m ↔	500x \equiv 25 μ m ↔	1000x \equiv 10 μ m ↔	

3.1.1 Steels and Iron Alloys

3.1.1.1 PM Steel Supermet Fe64 (Figures 7 and 8)

Specimen 46.5 / 450 / 8, vertical ⇒ Grain Boundary and Carbides etched, for DIC and Reflected Light o.k.

Specimen 66 / 415 / 8, vertical ⇒ Grain Boundary and Carbides etched, for DIC and Reflected Light o.k.

Specimen 75 / 270 / 8, vertical ⇒ Grain Boundary and Carbides etched, for DIC very suitable.

Note:

For the PM steels both the grain boundaries and also the different carbides were etched. The center of the etch mark is usually very suitable for normal micrographs enlarged up to 500 times. Towards the edge the etch intensity diminishes. Here high-contrast images can be produced with DIC. As this high-carbide content material is heterogeneously textured, a voltage of 5 keV suffices for optimal etching. Alternatively the etch time could be reduced.

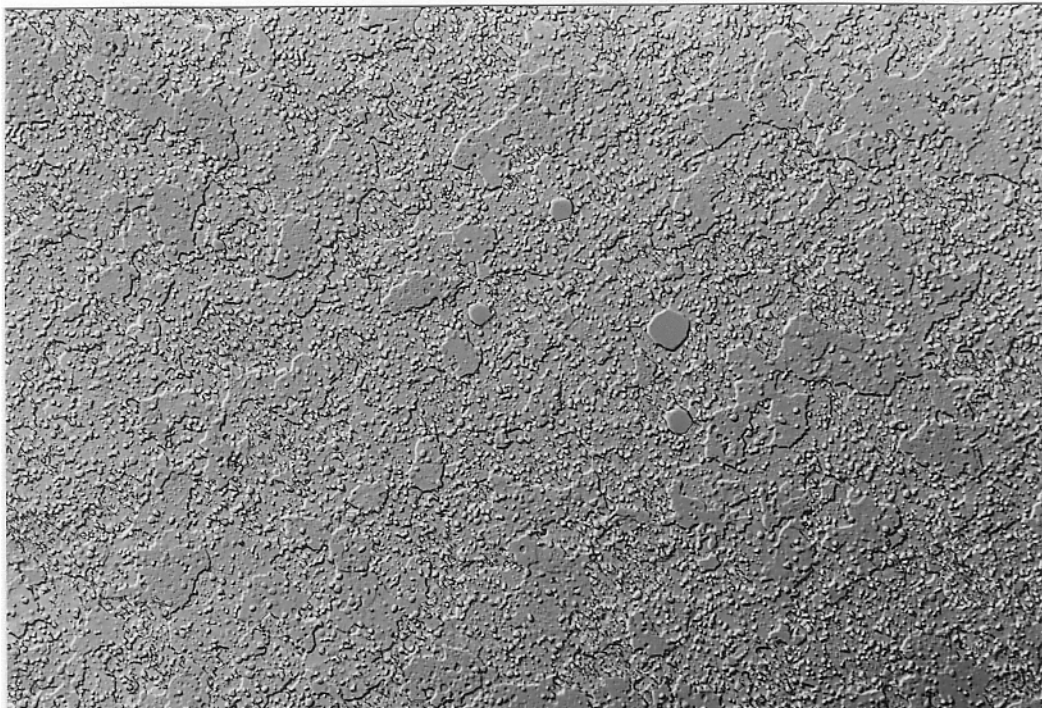
3.1.1.2 Tool Steel 100Cr6 (Figures 9 and 10)

Specimen 16.5 / 490 / 8, vertical ⇒ Grain Boundary and Carbides etched, for DIC and Reflected Light o.k.

Specimen 35 / 270 / 8, vertical ⇒ Grain Boundary and Carbides etched, for DIC very suitable.

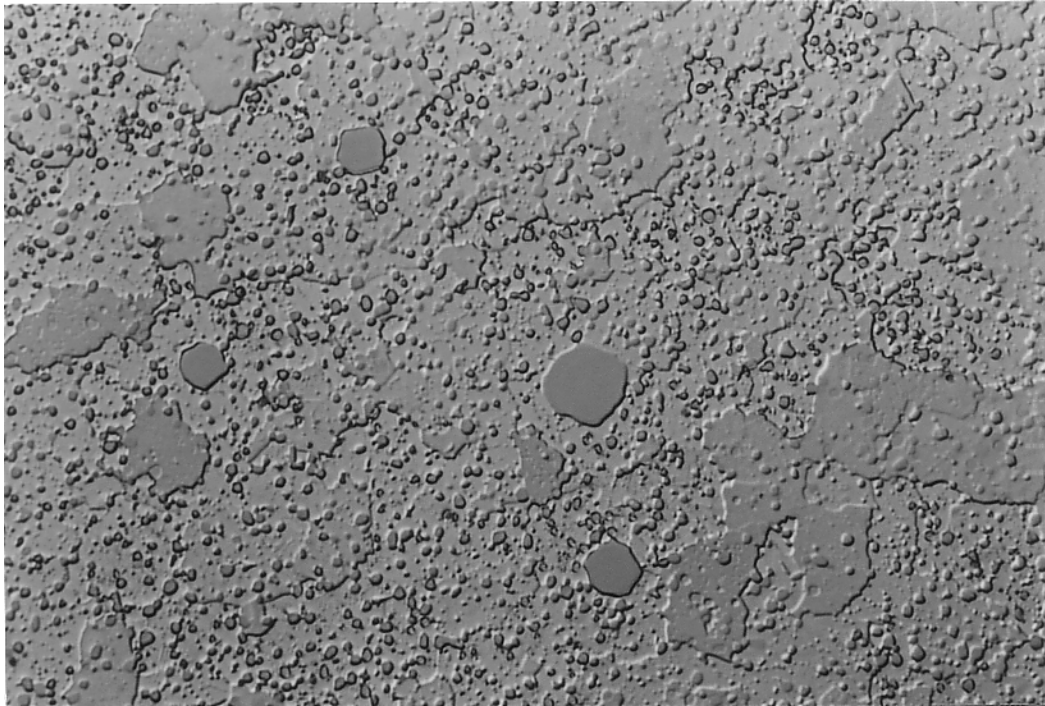
Note:

As the 100Cr6 steel is, similar to the PM steel, very heterogeneously textured, similarly a voltage of 5 keV or a shorter etch time suffices for optimal texture development.



500x

Figure 7: Texture of the PM Steel Supermet Fe64 (Specimen 4), Ion Beam Etched, Micrograph from the Edge of the Etch Mark, Parameter: 6.5kV / 450 μ A / 8mins



1000x

Figure 8: Texture of the PM Steel Supermet Fe64 (Specimen 4), Ion Beam Etched, Micrograph from the Edge of the Etch Mark, Parameter: 6.5 kV / 450 μ A / 8 mins, Sectional Enlargement of Figure 7



500x

Figure 9: Texture of the Tool Steel 100Cr6 (Specimen 1), Ion Beam Etched, Micrograph from the Edge of the Etch Mark, Parameter: 6.5 kV / 490 μ A / 8 mins

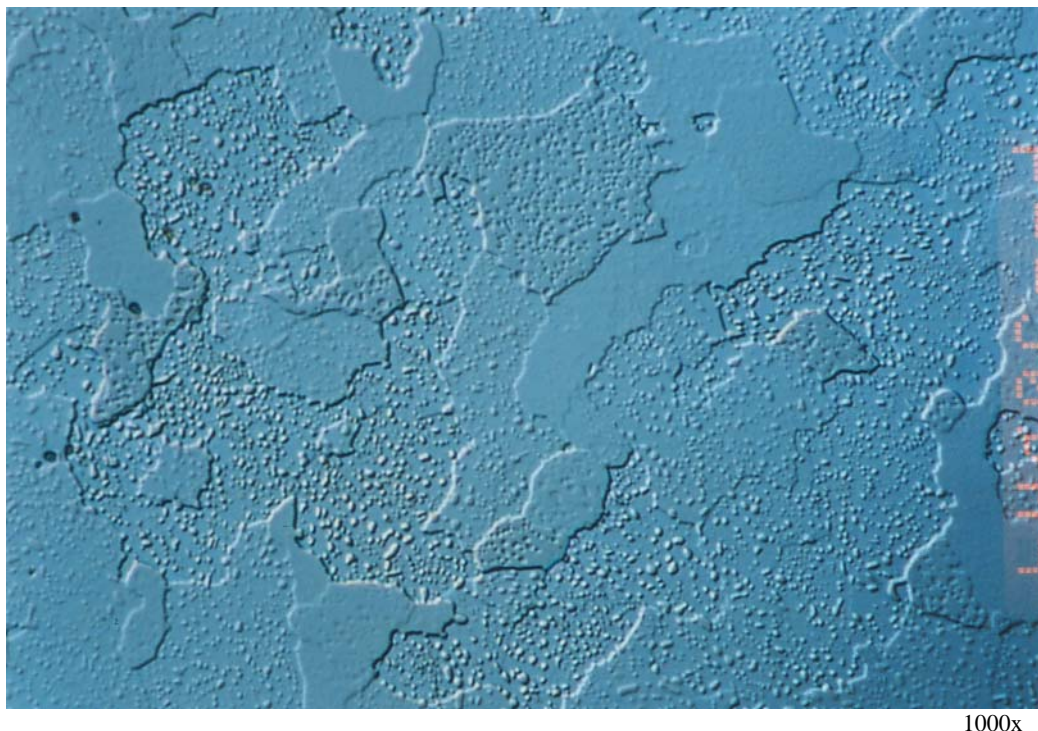


Figure 10: Texture of the Tool Steel 100Cr6 (Specimen 1), Ion Beam Etched, Micrograph from the Edge of the Etch Mark, Parameter: 6.5 kV / 490 μ A / 8 mins, Sectional Enlargement of Figure 9

3.1.1.3 Ferrite Stainless Steel X7Cr13 (Figures 11 and 12)

Specimen A/1 5 / 270 / 8, vertical \Rightarrow for DIC very suitable, o.k.

Specimen A/3 6 / 405 / 12, 45°, rotating \Rightarrow for DIC very good, carbides show up well.

Note:

No grave differences were ascertainable between vertical bombardment and bombardment below 45°. The grain surfaces were etched dependent upon orientation so that the grain boundaries are also clearly visible. Carbides are developed concisely at the grain boundaries and in part also on the grain surfaces (presumably at former grain boundaries).

3.1.1.4 FeCo Alloy (Figures 13 to 15)

Specimen 1 6 / 385 / 8, 52.5°, rotating \Rightarrow Grain Boundary slightly etched
 + 6 / 400 / 4, 45°, rotating \Rightarrow Grain Boundary strongly etched
 + 6 / 400 / 4, vertical, rotating \Rightarrow suitable for DIC and Reflected Light (up to 200x).

Specimen 3 6 / 430 / 8, vertical \Rightarrow for DIC and Reflected Light (up to 500x) o.k.
 etching stronger

Note: The grains of this FeCo alloy are of varying size, which comes out very well. As this alloy was subjected to annealing treatment, the specimens are surrounded by a layer of cinder. This was similarly textured by ion beam etching.



Figure 11: Texture of the Ferrite Stainless Steel X7Cr13 with Carbides at the Grain Boundaries, (Specimen A/3), Ion Beam Etched, Parameter: 6 kV / 405 μ A / 12 mins, 45°, Rotating

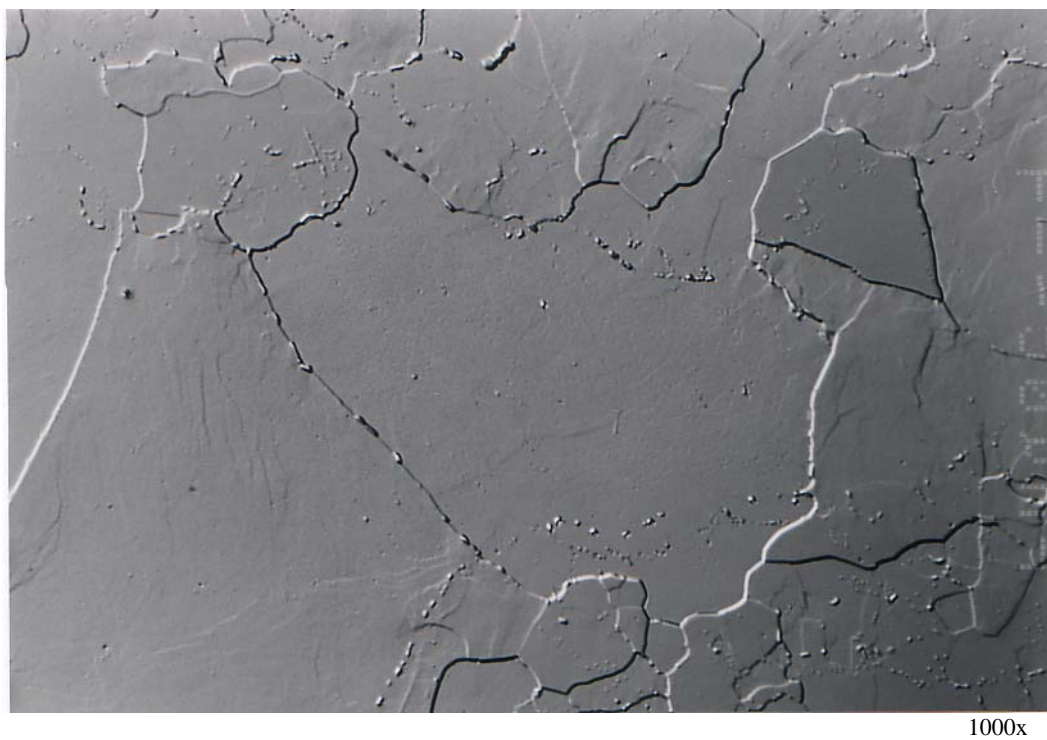
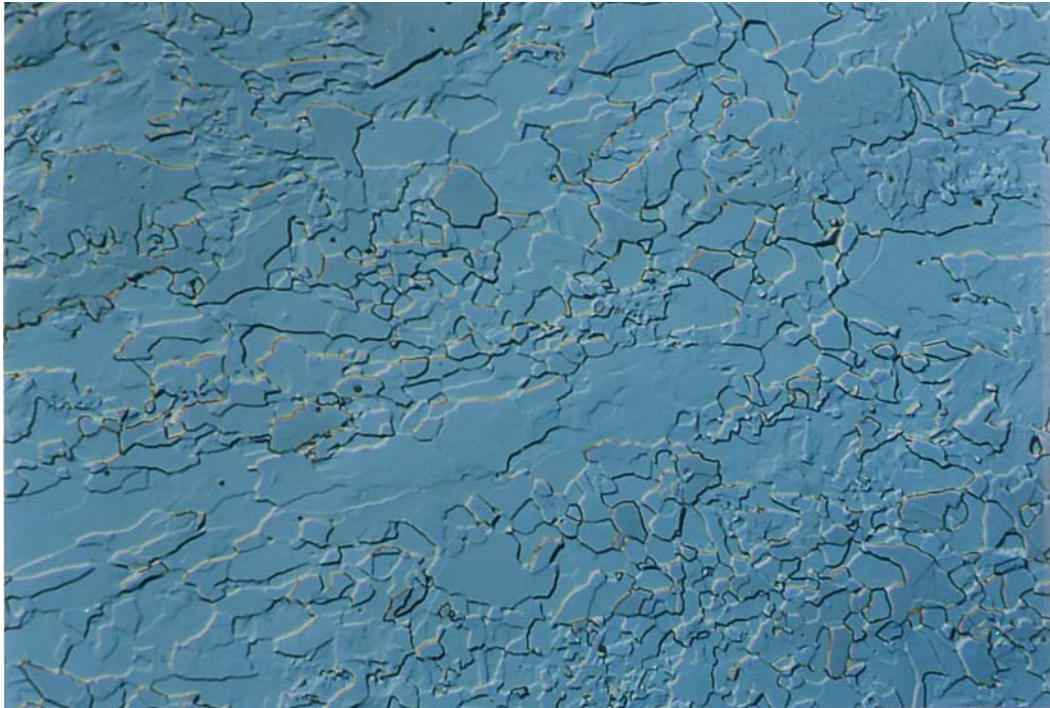
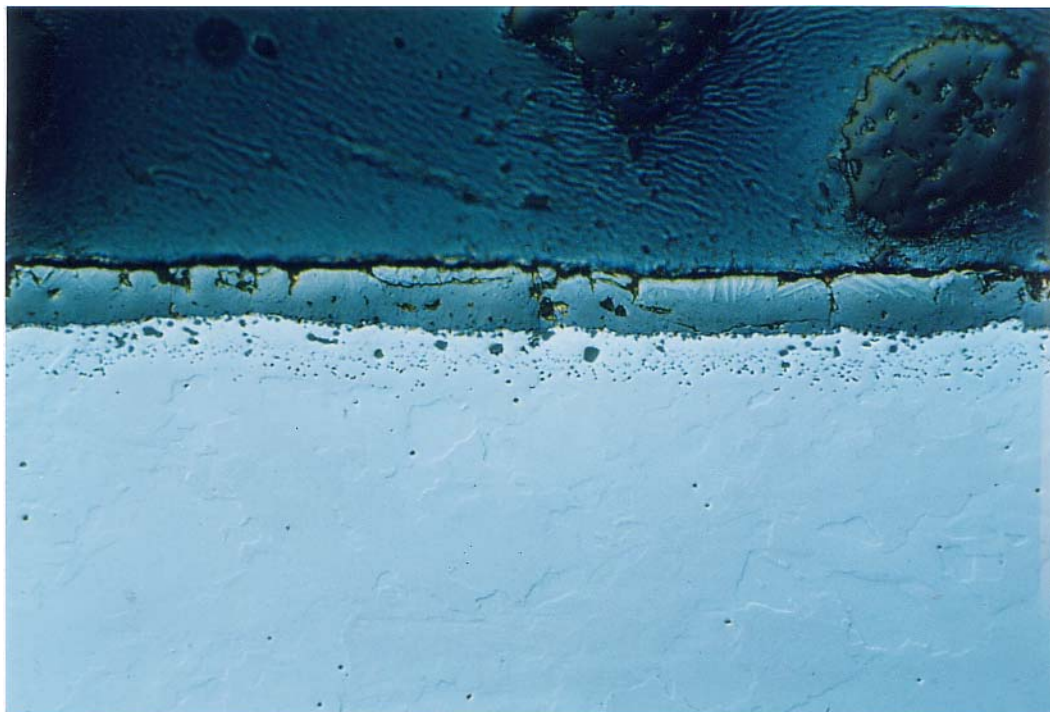


Figure 12: Texture of the Ferrite Stainless Steel X7Cr13 with Carbides at the Grain Boundaries, (Specimen A/3), Ion Beam Etched, Parameter: 6 kV / 405 μ A / 12 mins, 45°, Rotating, Sectional Enlargement of Figure 11



500x

Figure 13: Texture of the FeCo Alloy (Specimen 3), Ion Beam Etched, Micrograph of the Center of the Etch Mark, Parameter: 6kV / 430 μ A / 8min, Vertical, Rotating



500x

Figure 14: Layer of Cinder on the Annealed Specimen (Specimen 2) from FeCo Alloy, unetched

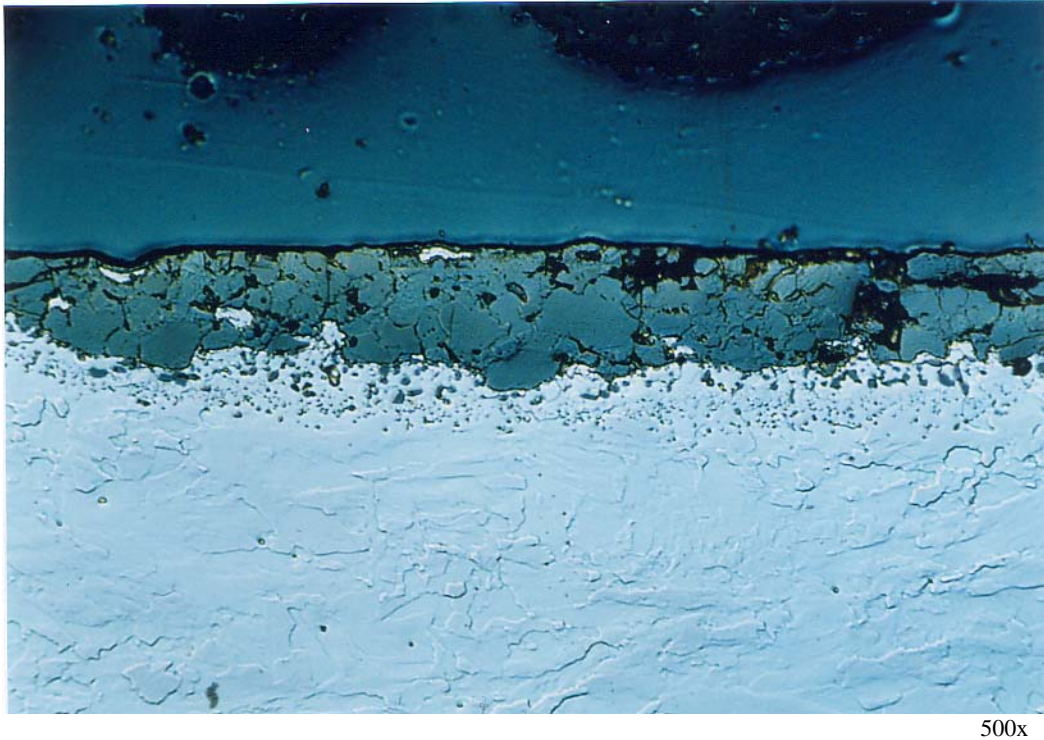


Figure 15: Layer of Cinder on the Annealed FeCo Alloy (Specimen 3), Ion Beam Etched, Parameter: 6 kV / 430 μ A / 8 mins, Vertical, Rotating

3.1.1.5 Hardened Steel Ck15

Specimen A/1 6 / 415 / 12, 45°, Rotating \Rightarrow for DIC very good, perlite slightly etched

Note:

The perlite is however not etched conventionally but only slightly textured.

3.1.1.6 Construction Steel Ck45 (ferrite /perlite) (Figures 16 to 18)

Specimen 2.4 3 / 100 / 16, vertical. (left) \Rightarrow fine texture development also of perlite
 + 4 / 150 / 8, vertical (right) \Rightarrow Texture (also perlite) is high-contrast
 $\Rightarrow\Rightarrow$ only specimen with optimal perlite etching

Specimen 2 6 / 405 / 6, 45°, rotating. \Rightarrow slight etching of the grain boundary, for DIC very suitable.
 + 6 / 405 / 4, vertical \Rightarrow Grain boundary etched, perlite also quite well textured, center for DIC too strong, for reflected light too weak, outside of center for DIC, however, suitable.

Specimen 5 6 / 405 / 8, vertical (high) \Rightarrow Almost too strong, for reflected light however o.k.

Specimen 6 6 / 405 / 12, 45°, rotating ⇒ Grain boundary not so strongly slanted as at under 30°, Perlite quite good, etching for DIC very good

Note:

Only once in the large number of experiments did a conventional texture development of perlite occur in the normal sense. That was the first etching after **the** equipment had been set up. Even a repeat run with the same parameters did not produce a typical perlite etching. Even the use of oxygen did not bring about the desired result. Nevertheless there must be etching conditions which as usual etch perlite.

3.1.1.7 Austenitic Steel 1.4301

Specimen 4/A 6 / 375 / 8, 10°, Rotating ⇒ Grain boundary etched, for DIC very good

Specimen 3/A 6 / 405 / 18, 60°, Rotating ⇒ fine etching of the grain boundaries
+ 6 / 405 / 4, vertical, rotating. ⇒ Etching for DIC and reflected light quite good

Note:

This austenitic steel revealed a great deal of deformation martensite, where it was not clear whether this had been caused by the preparation. From that point, the attempt was made to etch the surface using a low angle (60° to 65°). The result was the more often and the longer the specimen was etched the more unsatisfactory the surface became and artifacts appeared on it.



200x

Figure 16: Texture of the Construction Steel Ck45 (Specimen 2.4), Ion Beam Etched, Micrograph Taken between the Etch Points, Parameter: 3 kV / 100 μ A / 16 mins and 4 kV / 150 μ A / 8 mins, Perlite developed similarly as when using traditional chemical etch agents

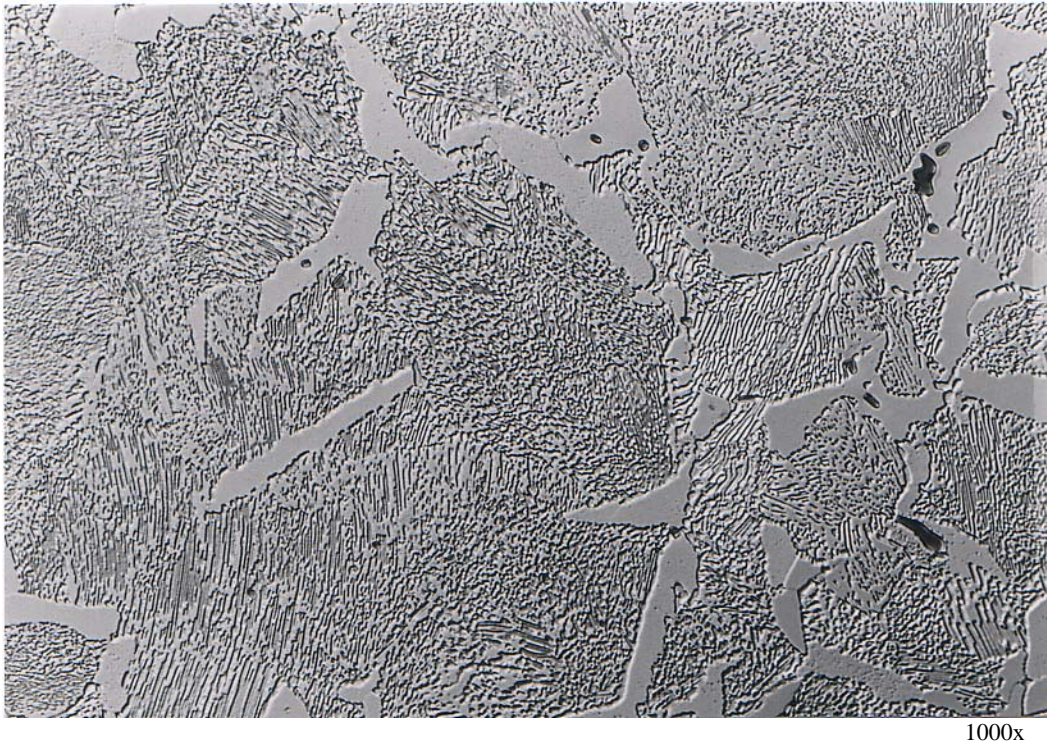


Figure 17: Texture of the Construction Steel Ck45 (Specimen 2.4), Ion Beam Etched, Micrograph Taken between the Etch Points, Parameter: 3 kV / 100 μ A / 16 mins and 4 kV / 150 μ A / 8 mins, Sectional Enlargement of Figure 16

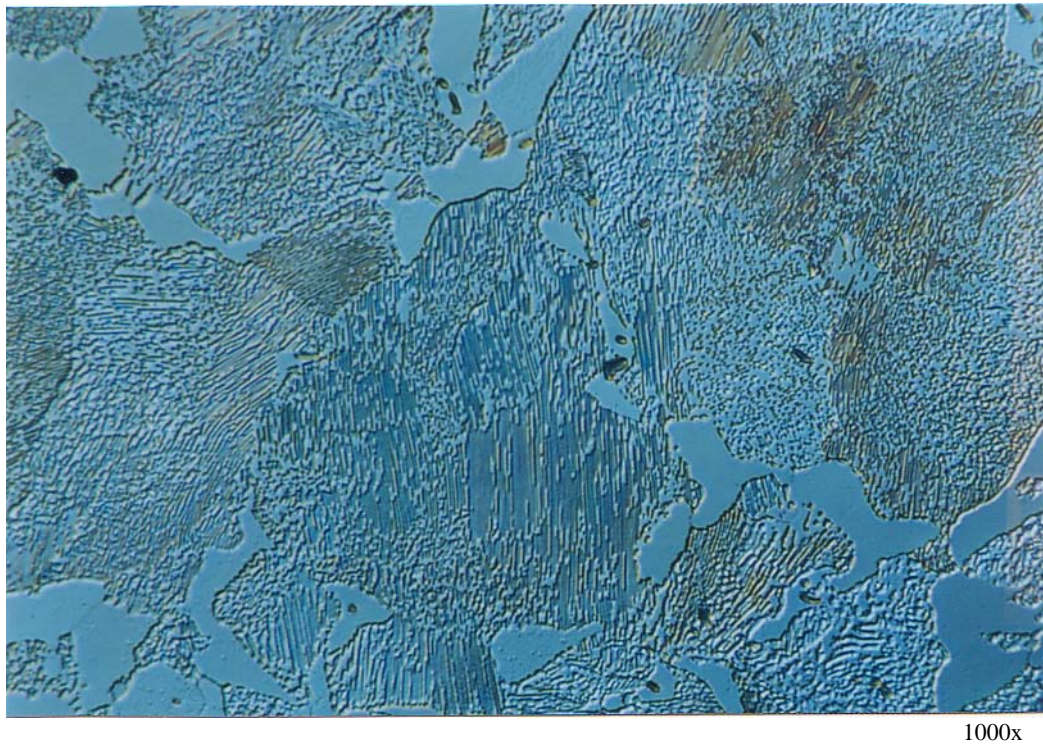


Figure 18: Texture of the Construction Steel Ck45 (Specimen 2.4), Ion Beam Etched, Micrograph Taken between the Etch Points, Parameter: 3 kV / 100 μ A / 16 mins and 4 kV / 150 μ A / 8 mins, similar Micrograph as for Figure 17, however in Color

3.1.2 Titanium and Titanium Alloys

3.1.2.1 High-Purity Titanium (Figures 19 to 21)

Specimen 1	6 / 405 / 8, vertical	⇒ for DIC and POL very good, o.k.
Specimen 3	6 / 395 / 12, 60°, rotating. ↓ Max at 520 μ A	⇒ Slight etching of the grain boundary and surface, acicular inclusions slightly etched and slanted, grains well visible with POL
	+ 6 / 395 / 8, vertical, ° rotating	⇒ more strongly etched for DIC and POL o.k.
	+ 6 / 395 / 7.2, vertical, ° rotating	⇒ for DIC and POL <u>very good</u> , for reflected light still not sufficient
Specimen 5	6 / 405 / 6, 65°, rotating	⇒ Grain boundary slightly etched, fine inclusions as well, partially slanted, in POL already contrastive
	+ 6 / 400 / 12, vertical, °, rotating	⇒ for reflected light almost sufficient
	+ 6 / 400 / 4, vertical, °, rotating	⇒ Grain surface smoothed, for DIC, POL and reflected light o.k.

Note:

Characteristic for Titanium materials is the fact that when observing them with polarized light the grains light up differently according to their orientation. The pre-requisite for this is sufficiently strong etching by ion bombardment. Usually the grain surfaces are then slightly rough to grainy. More detailed information on the inclusions in this material was not known.

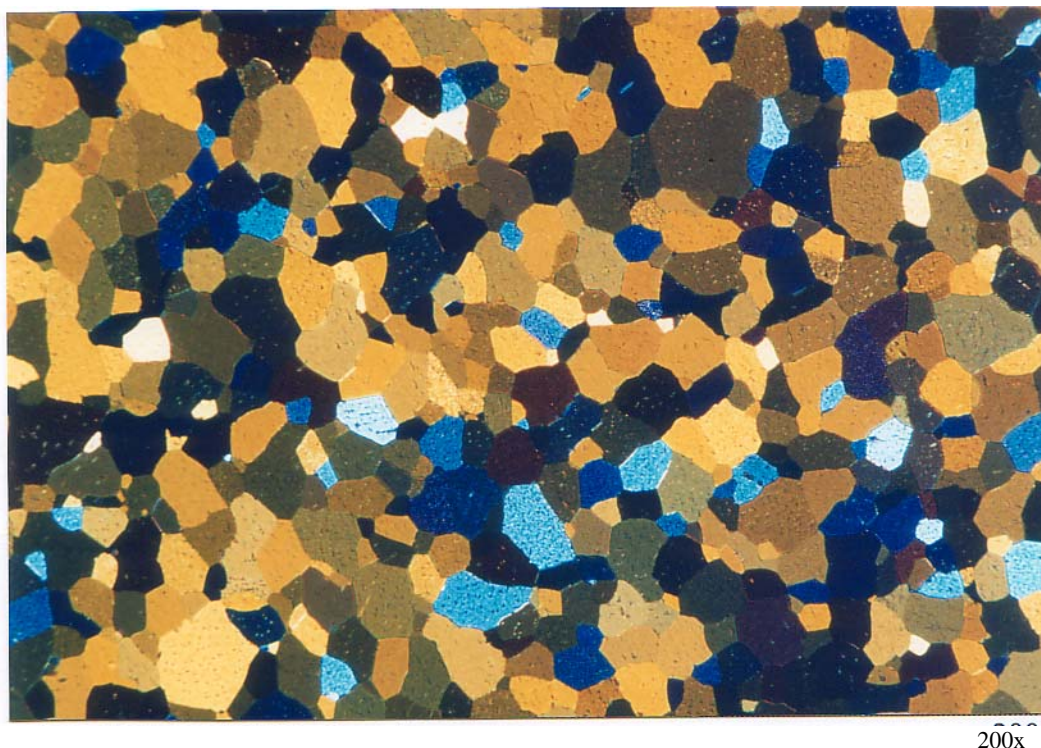


Figure 19: High-Purity Titanium Texture (Specimen 3), Ion Beam Etched, Micrograph with POL Filters, Parameter: 6 kV / 395 μ A / 12 mins, 65°, rotating
+ 6 kV / 395 μ A / altogether 15.2 mins, vertical, rotating

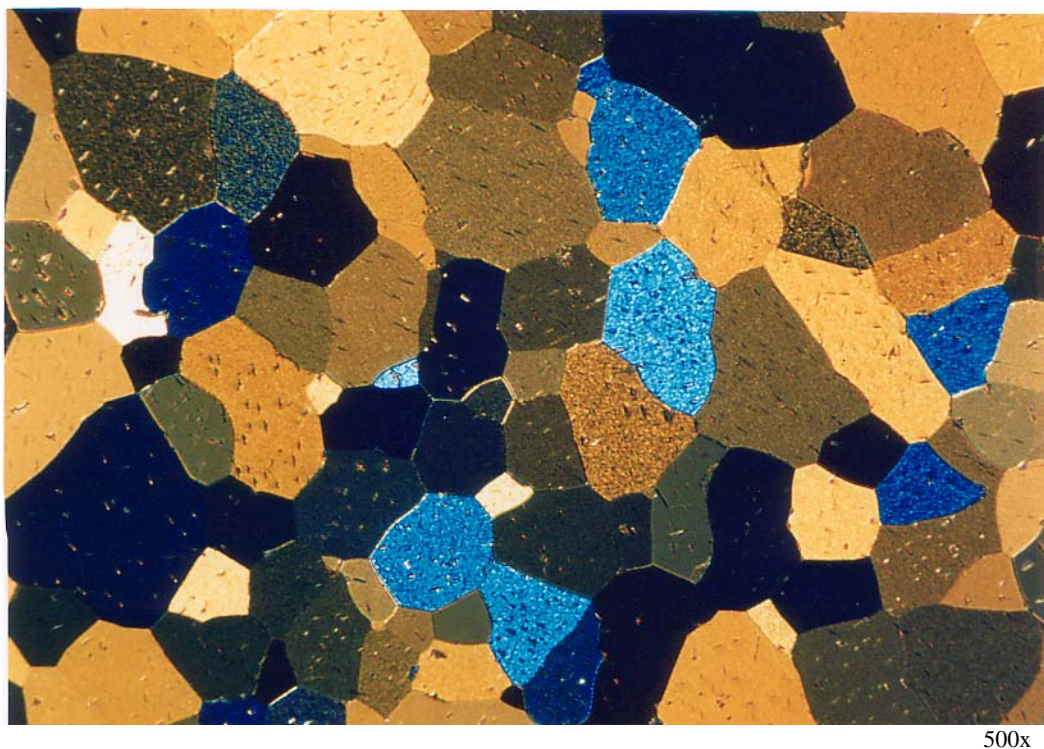


Figure 20: High-Purity Titanium Texture (Specimen 3), Ion Beam Etched, Micrograph with POL Filters, Similar Position as in Figure 19, Parameter: cf. Figure 19



Figure 21: High-Purity Titanium Texture (Specimen 3), Ion Beam Etched, Micrograph with DIC, practically the same position as Figure 20

3.1.2.2 Ti-Alloy with Si (Timetal 1100) (Figures 22 to 26)

Specimen 1 6 / 405 / 8, vertical. (point 1) ⇒ Center strongly etched, in POL very good o.k.

Specimen a 6.5 / 435 / 12, 65°, rot. ⇒ Ring-shaped etching at the outer edge of the
12mm high specimen, i.e. the etching angle was too low

+ 6.5 / 405 / 12, 52.5°, rotating ⇒ Silicides already visible, center of specimen hardly etched, Etching ring-shaped, here for DIC and POL o.k.

+ 6.5 / 435 / 4.4, vertical, rotating ⇒ Silicides disappear as a result of fiercer etching, for DIC, POL and reflected light well etched

Specimen b 6.5 / 435 / 16, 52.5°, rotating ⇒ Edge for DIC very good, silicides identifiable

+ 6 / 405 / 2.2, vertical, rotating ⇒ Etch point in the center of the specimen is very small, Silicides very clearly visible, altogether o.k.

Note:

This titanium alloy contains silicides which could not be determined with traditional chemical or electrochemical etching agents. These can be clearly identified with ion beam etching. However, the etching may not be too intensive nor too long, as then the silicides are smoothed or even etched out. The POL effect is only minimal or even non-existent for weaker etching.

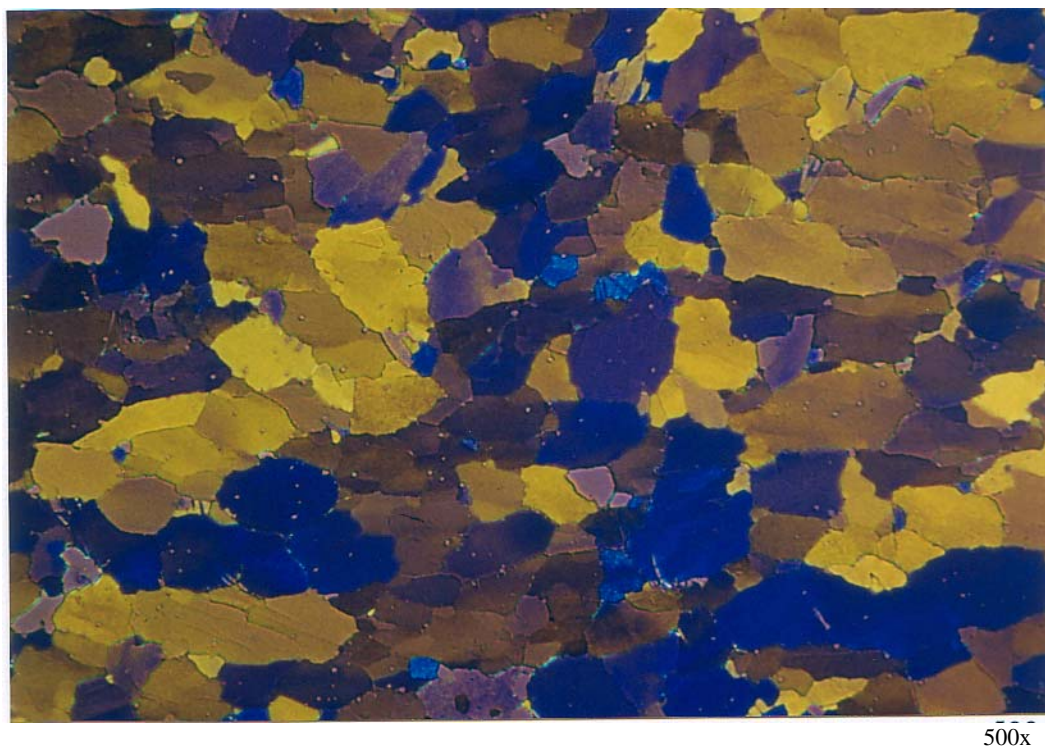
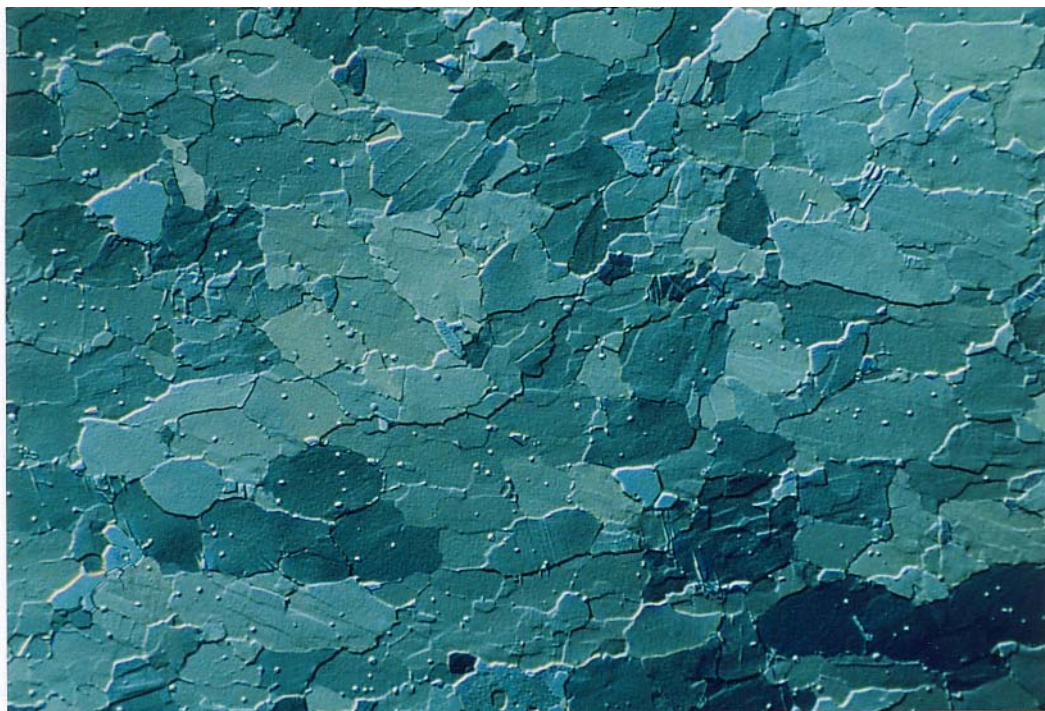
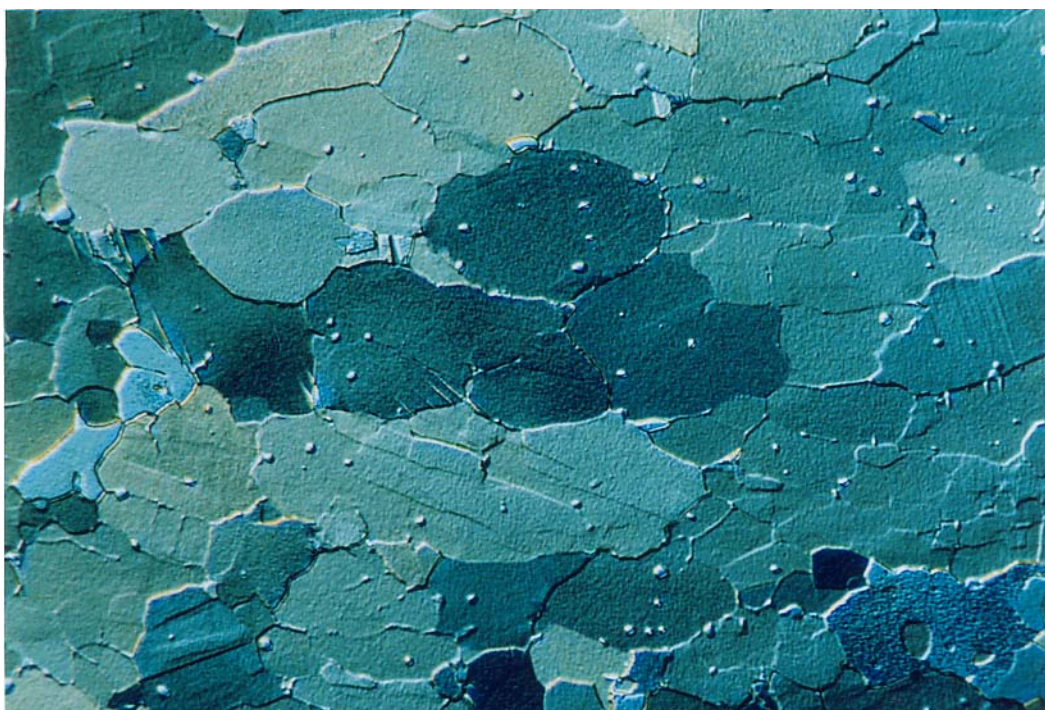


Figure 22: Texture of the Timetal 1100 Ti Alloy (Specimen 1), Ion Beam Etched (Point 1), Micrograph with POL Filters, Grains show Color Effects in accordance with their Orientation, Parameter: 6 kV / 405 μ A / 8 mins



500x

Figure 23: Texture of the Timetal 1100 Ti Alloy 1100 (Specimen 1), Ion Beam Etched (Point 1), Micrograph with DIC, the Same Position as for Figure 22, Fine Silicides are developed by Ion Beam Etching, Parameter: 6 kV / 405 μ A / 8 mins



1000x

Figure 24: Texture of the Timetal Ti Alloy 1100 (Specimen 1), Ion Beam Etched (point 1), Micrograph with DIC, Fine Silicides Are Developed by Ion Beam Etching, Parameter: 6 kV / 405 μ A / 8 mins, Section Enlargement of Figure 23

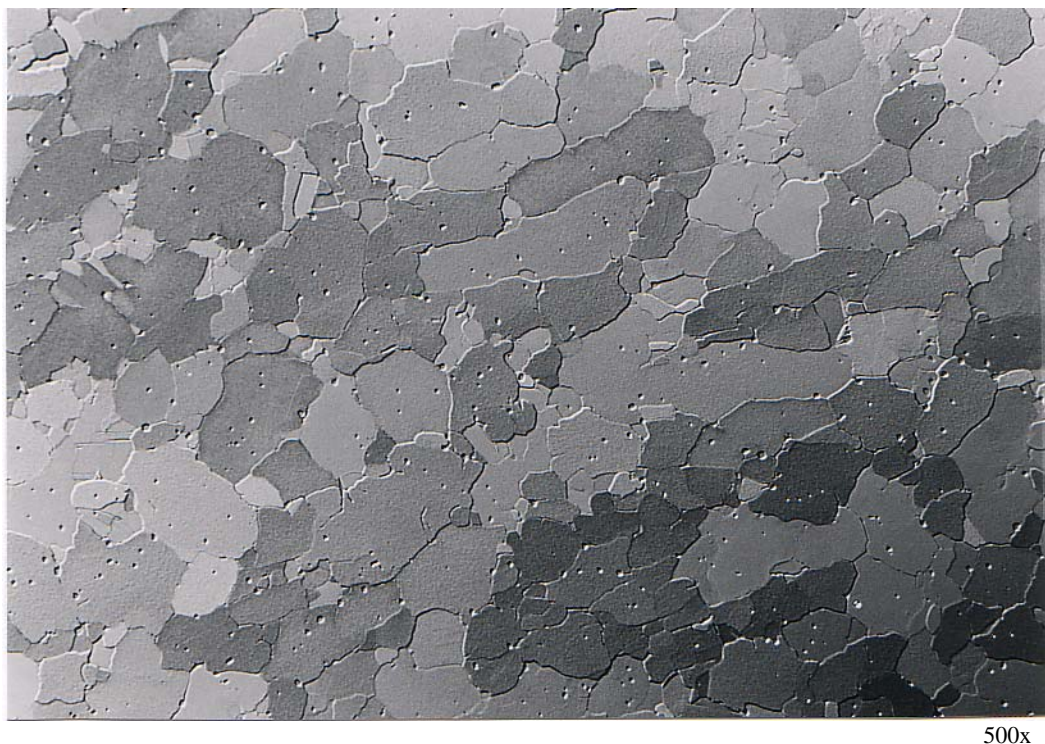


Figure 25: Texture of the Timetal Ti Alloy 1100 (Specimen **b**), Ion Beam Etched, Micrograph with DIC (etching relatively weak), Fine Silicides well visible, Parameter: 6.5 kV / 435 μ A / 16 mins, 52.5°, rotating + 6 kV / 405 μ A / 2.2 mins, vertical, rotating.

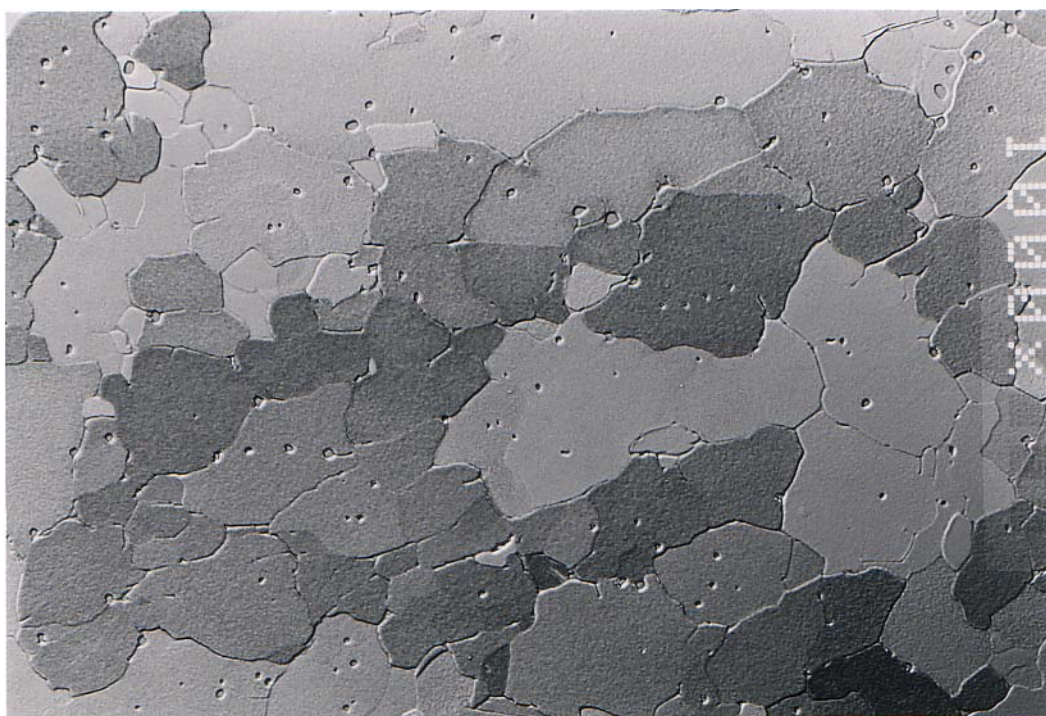


Figure 26: Texture of the Timetal Ti Alloy 1100 (Specimen **b**), Ion Beam Etched, Micrograph with DIC (etching relatively weak), Fine Silicides well visible, Parameter: 6.5 kV / 435 μ A / 16 mins, 52.5°, rotating + 6 kV / 405 μ A / 2.2 mins, vertical, rotating, Section Enlargement of Figure 25<

3.1.2.3 Alloy Ti6V4

Specimen 1 6 / 395 / 12, 60°, rotating. ⇒ Slight etching of the needle texture,
 E1907/106A ↓ Max at 444 μ A in POL however more easily recognizable
 + 6 / 415 / 8, vertical, rotating. ⇒ for DIC o.k., for reflected light not
 + 6 / 415 / 4, vertical, rotating. ⇒ for DIC and POL o.k., for reflected light not

Note:

The needle texture of this alloy can be imaged best using DIC.

3.1.3 Cu and Cu Alloys

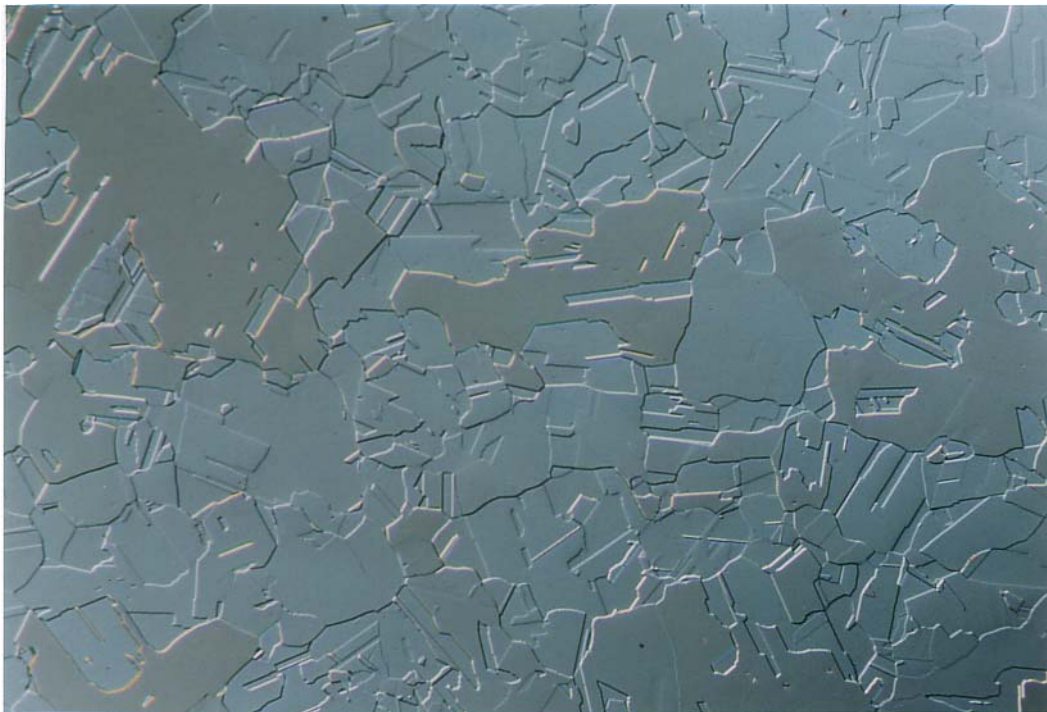
3.1.3.1 Cu Pole Pieces (High-purity-Cu, deformed and annealed with a tin coating (< 2 μ m) (Figures 27 to 30))

Specimen 2 4 / 170 / 6, vertical, rotating. ⇒ for DIC and reflected light o.k., also Sn coating o.k.

Specimen 4 6 / 370 / 2.4, vertical, rotating. ⇒ for DIC and reflected light optimal, also Sn coating o.k.

Note:

Both the copper and the Sn coatings are etched simultaneously which is not possible with traditional etching methods.



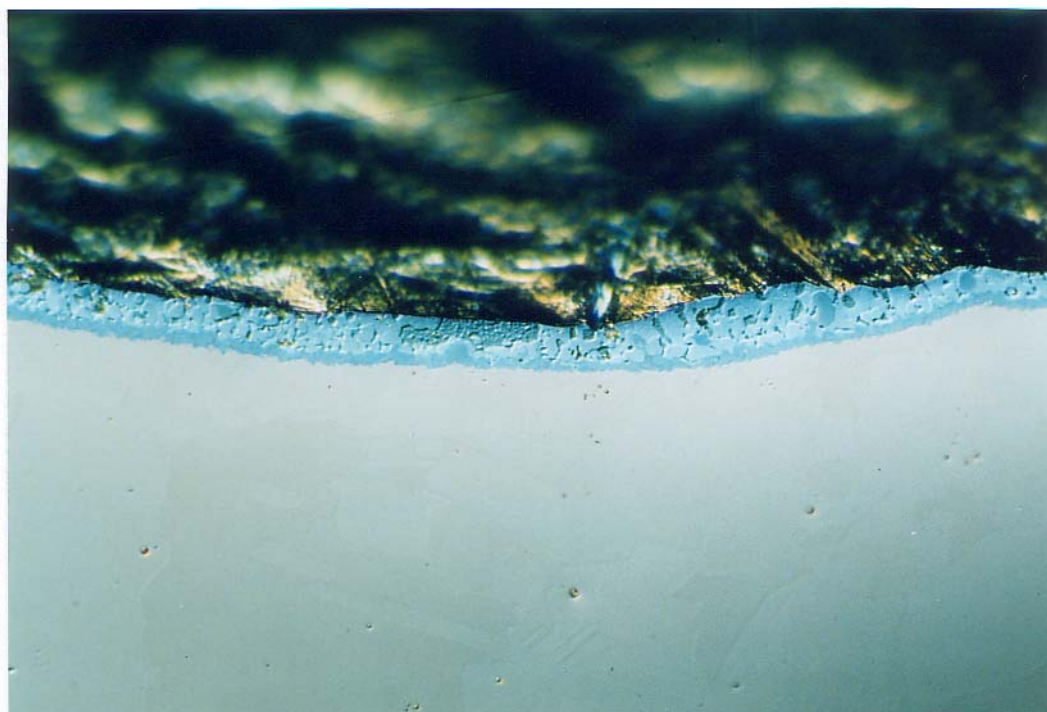
200x

Figure 27: Development of Grain Boundaries and Twins for high-purity-Cu by Ion Beam Etching (Pole pieces, Specimen 4), DIC Micrograph, Parameter: 6 kV / 370 μ A / 2.4 mins, vertical, rotating



500x

Figure 28: Development of Grain Boundaries and Twins for High-Purity Cu by Ion Beam Etching (Pole pieces, Specimen 4), DIC Micrograph, Section of Figure 27, Parameter: 6 kV / 370 μ A / 2.4 mins, vertical, rotating



500x

Figure 29: Sn Coating for Pole Pieces of High-Purity-Cu (Specimen 1), Edge in unetched state, DIC Micrograph

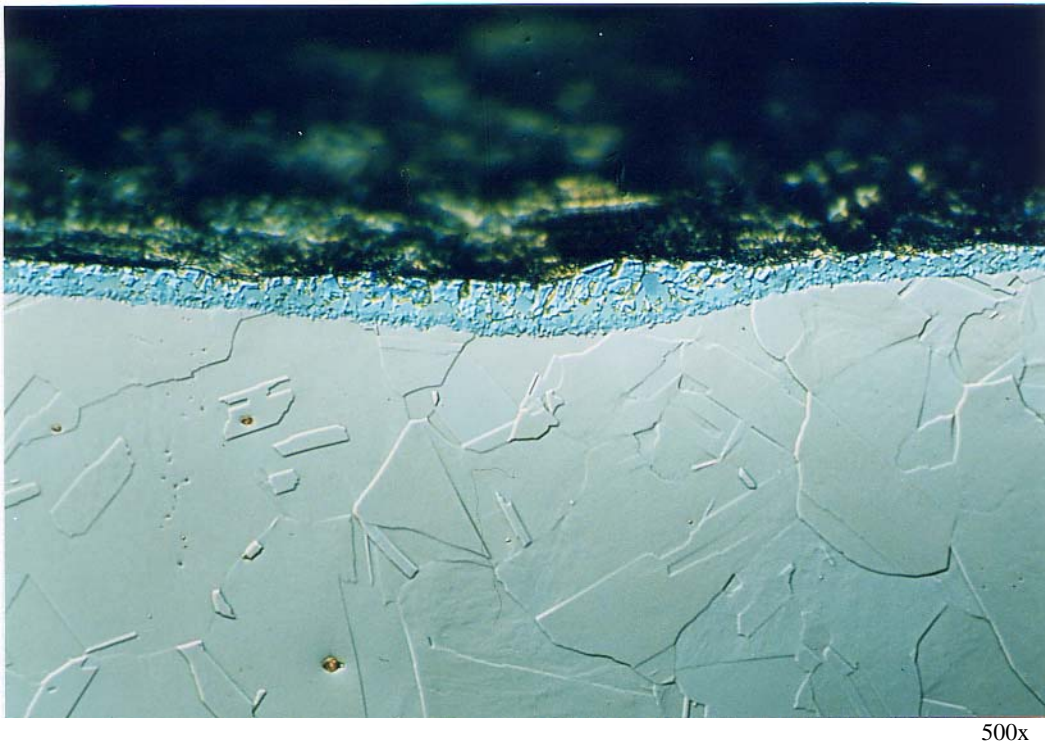


Figure 30: Sn Coating for Pole Pieces of High-Purity Cu after Ion Beam Etching (Specimen 2)
DIC Micrograph, Parameter: 4 kV / 170 μ A / 6 mins, vertical, rotating

3.1.3.2 Electrolyte Cu

Specimen 3 5 / 270 / 3.6, vertical \Rightarrow Grain boundary etching, very suitable for DIC and reflected light

Note:

As the sputtering yield (S) for copper is relatively high, short etching times are sufficient for the optimal structure development at a low acceleration voltage. Besides grain boundaries, deformations and glide bands are made visible.

3.1.3.3 CuCrZr Alloy, Material 2.1293

Specimen 1 6 / 405 / 0.4, 65°, rotating \Rightarrow no real change
 + 6 / 415 / 3.4, vertical, rotating. \Rightarrow already well etched but insufficiently
 + 6 / 415 / 0.4, vertical, rotating. \Rightarrow for DIC and reflected light o.k.

Specimen 2 6 / 425 / 3.4 (*Specimen off center*) \Rightarrow one half etched, o.k.
 + 6 / 405 / 2.2 (*Specimen off center*) \Rightarrow other half etched, o.k.

Note:

This alloy contains light blue inclusions which are preserved with weak etching, with stronger etching they are dissolved. Deformations in the material are visible.

3.1.3.4 Machine Brass: CuZn39Pb3 (Figures 31 und 32)

Specimen A1 6 / 425 / 0.4, 65°, rotating ⇒ slight etch attack of α -Mk and Pb-droplets
 + 6 / 425 / 2.4, vertical ⇒ optimum large etch mark, for DIC and reflected light o.k.!

Specimen 2a 6 / 405 / 0.4, 65°, rotating ⇒ Pb-droplets cleaned, α -Mk slightly etched,
 8 mm high Center somewhat more strongly etched, as
low angle specimen
 + 6 / 405 / 1.2, vertical, rotating. ⇒ etch mark in size o.k., optimal etching
 for DIC micrographs

Note:

Lead is alloyed to the machine brass so that in the mechanical processing (e.g. lathe tooling), a short chip is created. When etching with chemical agents, the lead droplets are dissolved. Ion etching does not by contrast cause this.

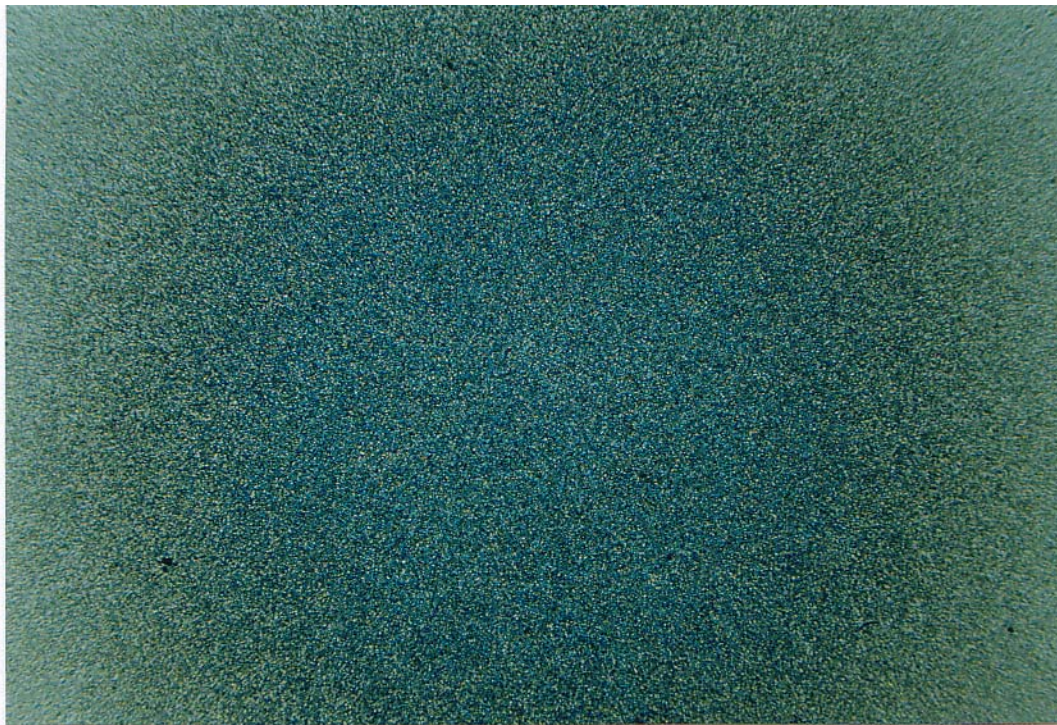
3.1.3.5 Composite Wire CuNiAg

Specimen 1 (longitudinal) 6 / 365 / 0.4, 45°, rotating ⇒ cleaned, slight etching of three components

Specimen 2 (transverse) 6 / 365 / 1, 45°, rotating ⇒ slight etching of three components

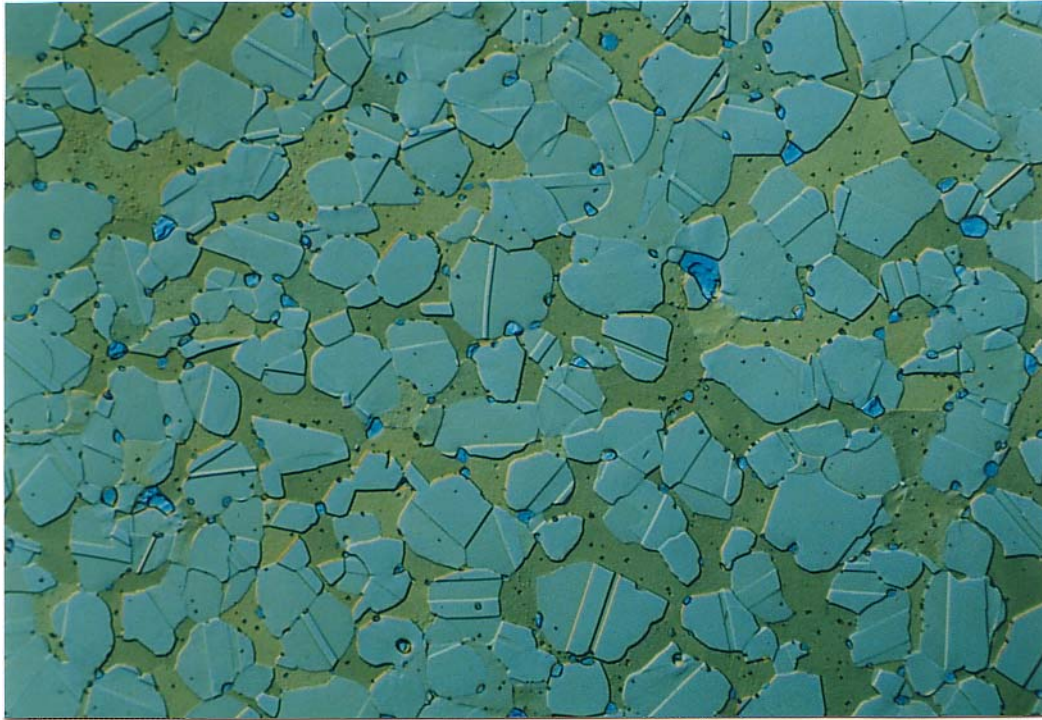
Note:

All etch tests with chemical and electrochemical agents were unsuccessful. The structural features of all three components could only be developed optimally by ion beam etching.



12.5x

Figure 31: Etch Mark for Machine Brass by means of Ion Bombardment (Specimen 2a),
 Parameter: 6 kV / 405 μ A / 0.4 mins, 65°, rot + 6 kV / 405 μ A / 1.2 mins, vertical, rotating.



500x

Figure 32: Structure Development for Machine Brass by means of Ion Beam Etching (Specimen 2a), DIC Micrograph, Lead Droplets Retained, in part Twins visible in Lead Droplets Parameter: 6 kV / 405 μ A / 0.4 mins, 65°, rot + 6 kV / 405 μ A / 1.2 mins, vertical, rotating.

3.1.4 Al and Al Alloys

3.1.4.1 High-Purity Aluminium (Figures 33 to 38)

Specimen 1	6 / 405 / 12.4, 45°, rotating	⇒ Slight etching of the grain boundaries
	+ 6 / 405 / 6, 45°, rotating	⇒ Grain boundaries better, slight POL effect
	+ 6 / 365 / 6, 45°, rotating	⇒ better POL effect
	+ 6 / 405 / 6, 45°, rotating	⇒ POL effect somewhat strengthened
	+ 6 / 395 / 12, 65°, rotating	⇒ POL effect of certain grains stronger

Specimen 46	405 / 12, 52.5°, rotating	⇒ already quite good, grain boundary etched, grain surface rough to grainy
	+ 6 / 405 / 8.2, 45°, rotating	⇒ Grain boundary much better
	+ 6 / 405 / 4, 45°, rotating	⇒ to this extent o.k.
	+ 6 / 405 / 6, 45°, rotating	⇒ POL effect stronger, grain surface rough to grainy
	+ 6 / 405 / 5.4, 45°, rotating	⇒ very good POL effect, grain surface more strongly rough to grainy.

Note:

High-purity Al can only be etched, to judge from the tests, at an angle of about 45°. The grain boundaries are slanted analogously to the incident angle of the ions. A number of grains (not all) get from the ion bombardment, depending on their orientation, a rough, sometimes grooved surface, whereby a color effect is created when observing the sample with polarized light.



Figure 33: Structure of High-Purity-Aluminium after Ion Beam Etching (Specimen 4), POL Micrograph (sufficient rough to grainy grain surfaces appear in color),
Parameter: 6 kV / 405 μ A / 12 mins, 52,5°, rotating
+ 6 kV / 405 μ A / in total 24 mins, 45°, rotating

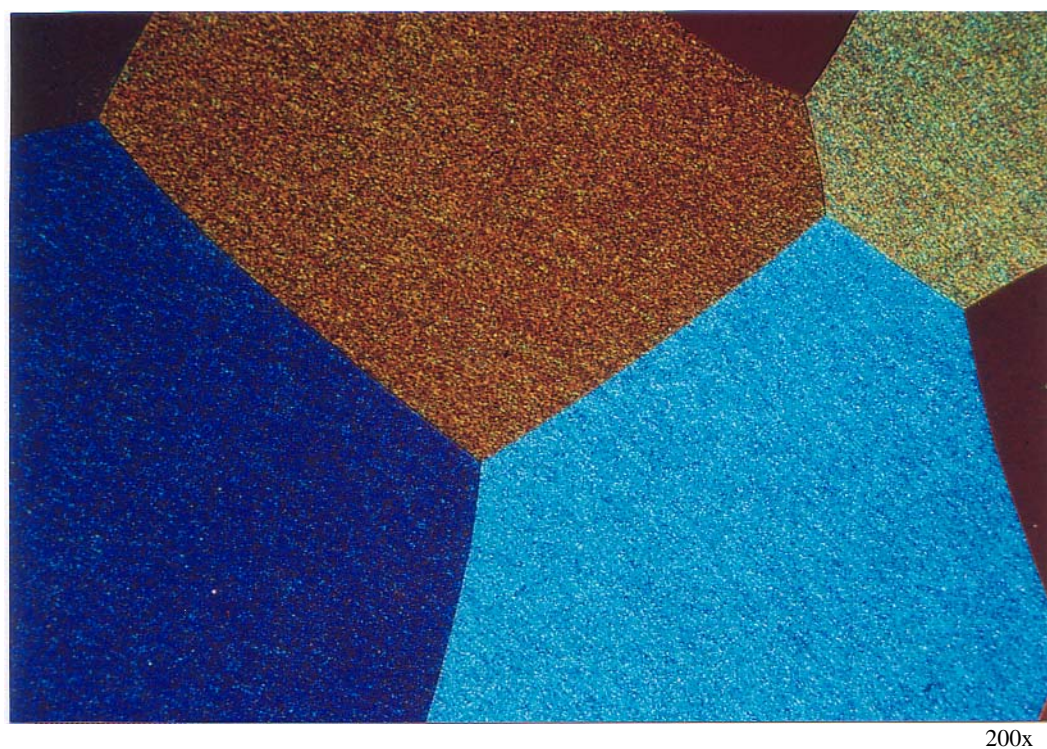
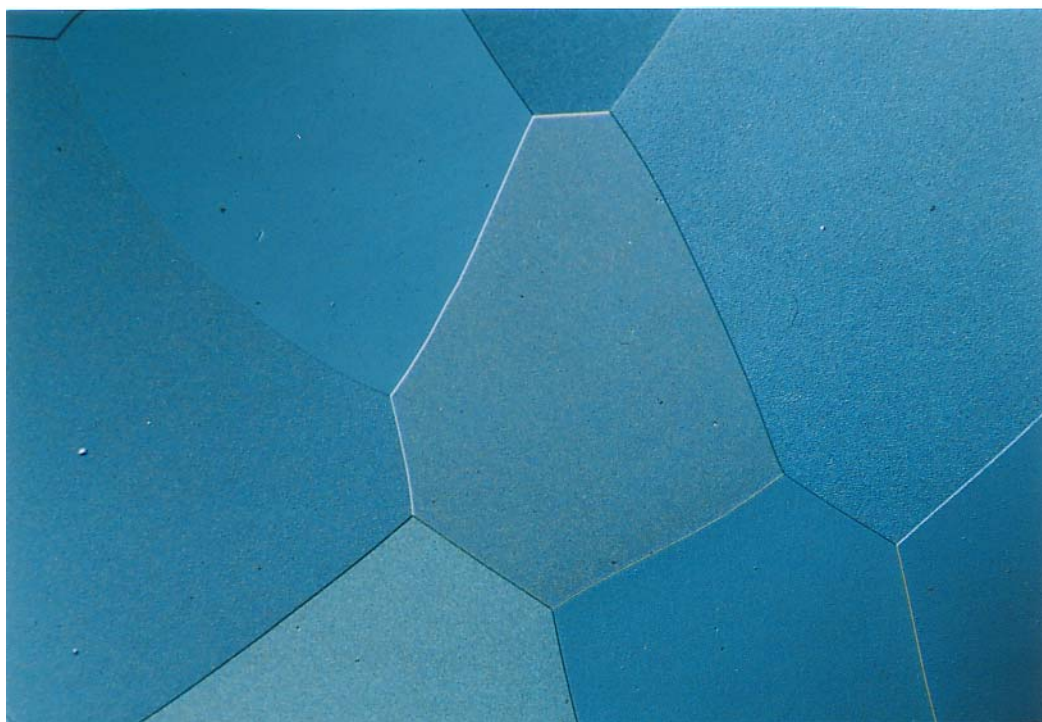
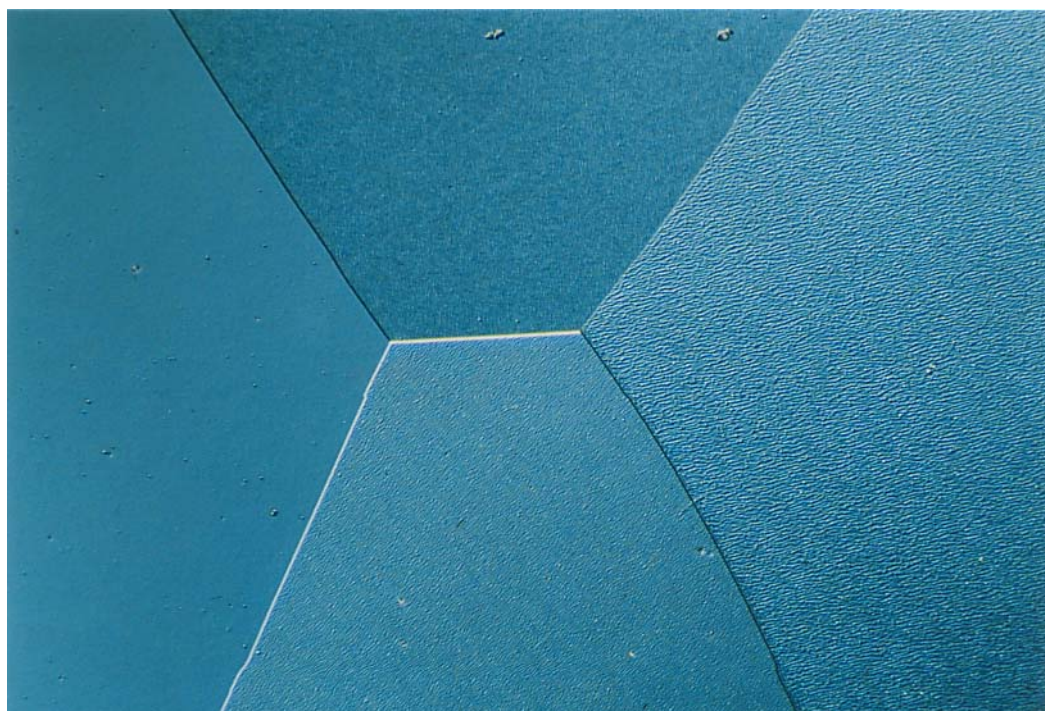


Figure 34: Structure of High-Purity-Aluminium after Ion Beam Etching (Specimen 4), POL Micrograph, Enlarged Section of Figure 33



200x

Figure 35: Structure of High-Purity-Aluminium after Ion Beam Etching (Specimen 4), DIC Micrograph, Same Position as in Figure 34,
Parameter: 6kV / 405 μ A / 12mins, 52,5°, rotating
+ 6kV / 405 μ A / in total 24mins, 45°, rotating



500x

Figure 36: Structure of High-Purity-Aluminium after Ion Beam Etching Specimen 4), DIC Micrograph, Section of Figure 35

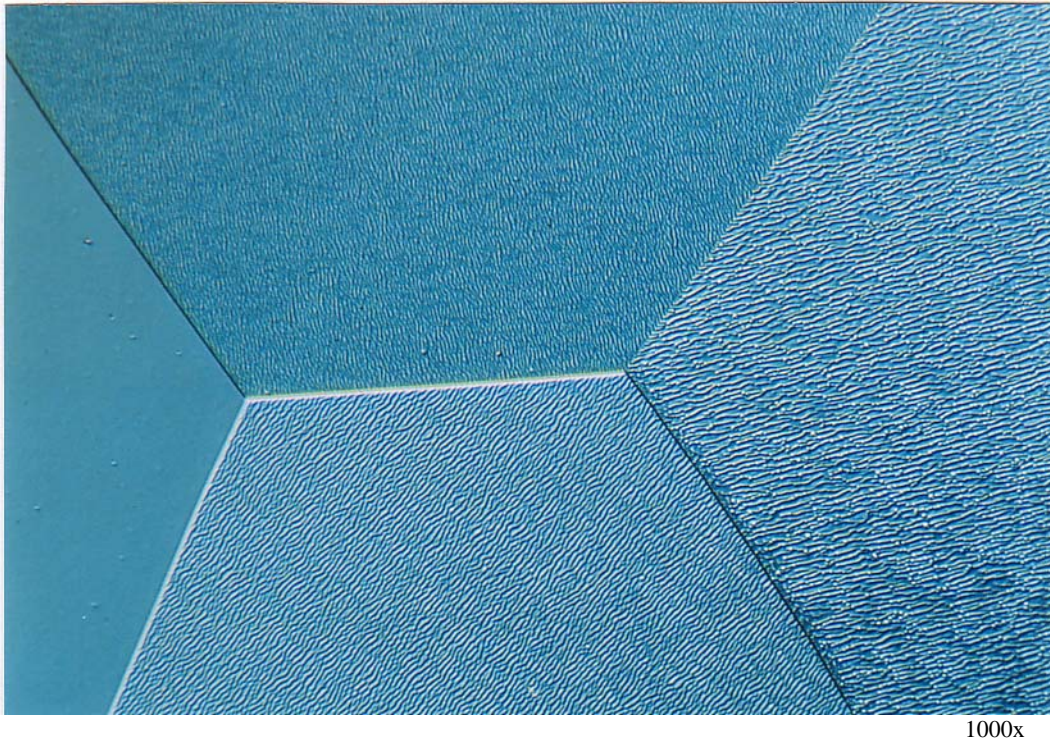


Figure 37: Structure of High-Purity-Aluminium after Ion Beam Etching (Specimen 4), DIC Micrograph, Section of Figure 36,
 Parameter: 6 kV / 405 μ A / 12 mins, 52,5°, rotating
 + 6 kV / 405 μ A / in total 24 mins, 45°, rotating



Figure 38: Structure of High-Purity-Aluminium after Ion Beam Etching (Specimen 4), DIC Micrograph, Section of Figure 35
3.1.4.2 Machining Alloy: AlMgSiPb / F28 (Figure 39)

old specimen	6 / 400 / 8,vertical + 4 / 120 / 8,vertical	⇒ mark and etching very good, o.k. ⇒ <i>new point</i> , very weak etching
Specimen II/2	6 / 415 / 8,vertical	⇒ Etch mark in terms of size o.k., edge suitable for DIC
Specimen 2.2	6 / 404 / 1, 75°, rotating + 6 / 405 / 8,vertical	⇒ Difficult to judge ⇒ Diameter of the etch mark o.k., edge good for DIC
Specimen 2.5	6.5 / 450 / 8,vertical	⇒ Mark very small, in the center strongly over etched → new
Specimen 2.5	6 / 405 / 6, 65°, rotating 6 / 405 / 6, vertical, rotating.	⇒ Pb-inclusions slanted, slight polish effect ⇒ Center strongly etched, edge <u>optimal for DIC</u> , o.k.

Note:

This machining alloy similarly contains lead for better machining. With ion beam etching the Pb inclusions are retained with simultaneous development of the grain boundaries.



1000x

Figure 39: Structure of the Machining Alloy AlMgSiPb after Ion Beam Etching (old specimen), Micrograph of edge of the etch mark with DIC, Lead droplets are retained in addition to other intermetallic phases, Parameter: 6 kV / 400 μ A / 8 mins, vertical and 4 kV / 120 μ A / 8 mins, vertical

3.1.4.3 Casting Alloy AlMg3 (non-hardenable)

Specimen 3 (IV)	6 / 405 / 10, 65°, rotating + 6 / 405 3.4, vertical, rotating	⇒ Grain boundaries already clearly etched, dark colored, inclusions thus far o.k. ⇒ optimal etching for micrographs in DIC
Specimen 4a (IV)	6.5 / 385 / 6, 50°, rotating	⇒ Grain boundaries etched, grain surfaces Smooth (some grain surfaces however reveal texture), inclusions o.k., center more strongly etched
Specimen 5a (IV)	6 / 395 / 4, 45°, rot + 6 / 380 / 2, 50°, rot + 6 / 405 / 4, 15°, rotating	⇒ Grain boundaries finely etched, Inclusions thus far o.k. ⇒ Grain boundaries more strongly etched towards the edge, in the specimen center less etching, inclusions acceptable ⇒ Grain boundaries more strongly etched, precipitates at the grain boundaries.

Note:

For this Al casting alloy AlMg3, besides the grain boundaries, the various intermetallic combinations (described above as inclusions) are etched. However, most of them are rapidly over-etched and even perhaps dissolved. Therefore the etch attack should be as mild as possible and so performed that the contrast for DIC micrographs is sufficient. For ion bombardment with a steep angle, fine point-like precipitates form at grain boundaries whereby it is not certain whether these are merely artifacts.

3.1.4.4 Casting Alloy AlMg4.5Mn (non-hardenable) (Figures 40 and 41)

Specimen 1	6 / 405 / 10, 65°, rotating + 6 / 405 / 3.4, vertical, rotating + 6 / 405 / 6, 55°, rotating + 6 / 405 / 6, 55°, rotating	⇒ grain surfaces slightly roughened ⇒ Grain boundaries only indicated, inclusions more intensely picked out ⇒ slight etching of the grain boundaries, inclusions thus far o.k. ⇒ Grain boundaries etched, inclusions slanted grain surfaces lightly roughened
Specimen 3	6 / 405 / 1, 55°, rotating (<i>angle in the interim changed</i>) + 6 / 405 / 10, 52.5, rotating + 6 / 405 / 16, 45°, rotating + 6 / 405 / 16, 30°, rotating + 6 / 415 / 6, 10°, rotating	⇒ ring-shaped fine etching of the grain boundaries and the inclusions, formation of precipitates at the grain boundaries ⇒ ring-shaped slight etching of the grain boundaries, inclusions relief-like, however thus far o.k. ⇒ no important changes, etch or polish effect ring-shaped, specimen center tarnished ⇒ small etch mark in the center of the

specimen, presumably during the etching precipitates for at grain boundaries, in the center slight segregation in the grains.

Note:

A phenomenon was observed in particular for Specimen 3: The grain boundaries can only be etched with difficulty. Instead fine precipitates occur at the grain boundaries with progressive ion bombardment. Moreover a change occurred within the grains, a coloring of the grain surfaces took place which may possibly be linked to segregation. What must have taken place quasi was heat treatment. Possibly the heat dissipation was insufficient as the specimen was embedded. The intermetallic combinations (designated above as inclusions) were to that extent acceptable.



200x

Figure 40: Texture of the Al Casting Alloy AlMg4.5Mn after Ion Beam Etching (Specimen 1), Micrograph with DIC, Grain Boundaries and Intermetallic Combinations Etched,

Parameter: 6kV / 405 μ A / 10mins, 65°, rotating,
 + 6kV / 405 μ A / 3.4mins, vertical, rotating
 + 6kV / 405 μ A / in total 12mins, 55°, rotating

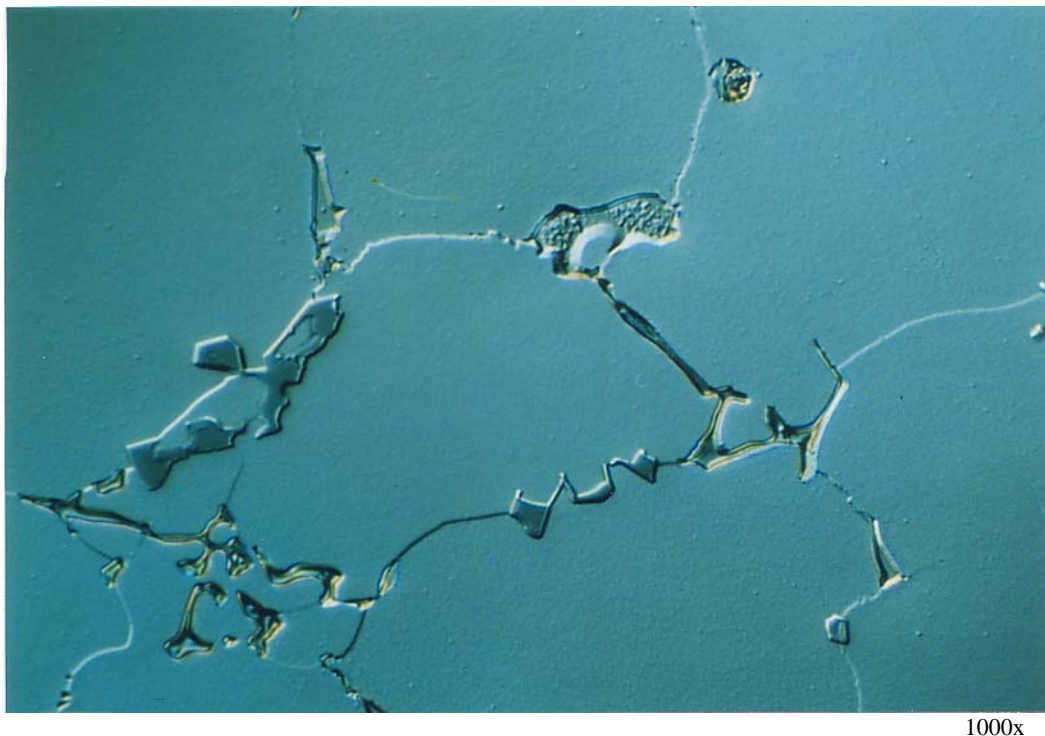


Figure 41: Texture of the Al Casting Alloy AlMg4.5Mn after Ion Beam Etching (Specimen 1), Micrograph with DIC, Enlarged Section of Figure 40

3.1.4.5 AlMg4.5Mn/15G, Annealed and Rolled, Strained Texture (non-hardenable) (Figures 42 to 44)

- | | |
|--|--|
| Specimen 4 6 / 405 / 6.4, 45°, rotating
(old specimen) | ⇒ Grain boundaries and grain surfaces (here precipitates not quite as strong) etched, intermetallic phases o.k. |
| Specimen 4a 6 / 385 / 6, 50°, rotating
+ 6 / 405 / 4.2, 15°, rotating | ⇒ Grain boundaries etched, grain surfaces slightly rough to grainy
⇒ string of pearls or precipitates at grain boundaries |
| Specimen 5 6 / 405 / 8, 55°, rotating
(old specimen) | ⇒ Grain boundaries and grain surfaces etched (here precipitates in part coarser), intermetallic phases o.k. |
| Specimen 6 6 / 395 / 6.4, 45°, rotating
(old specimen) | ⇒ Grain boundaries and grain surfaces etched (precipitates however not quite so strong), intermetallic phases o.k. |

Note:

In the tests performed with this alloy, the impact of the incident angle on the texture formation is particularly intense. At a low incident angle (55° to 65°), the grains surfaces are etched rough to grainy, in addition to a slight grain boundary etching, or precipitates appear to be strongly etched. For a medium angle (about 45°), the grain boundaries are etched better

and the grain surfaces relatively smooth. A relatively steep angle ($< 22.5^\circ$) means that precipitates in the form of strings of pearls can be formed at the grain boundaries. To what extent these are artifacts still has to be clarified. The intermetallic phases remain acceptable.



500x

Figure 42: Texture of the Rolled Al Alloy AlMg4.5Mn/15G after Ion Beam Etching (Specimen 6), Micrograph with DIC, Grain Boundaries Developed, Grain Surfaces Relatively Smooth, Parameter: 6kV / 405 μ A / 6.4mins, 45 $^\circ$, rotating



500x

Figure 43: Texture of the Rolled Al Alloy AlMg4.5Mn/15G after Ion Beam Etching (Specimen 5), Micrograph with DIC, Grain Boundaries Developed, Grain Surfaces Rough to Grainy, Parameter: 6kV / 405 μ A / 8mins, 55 $^\circ$, rotating

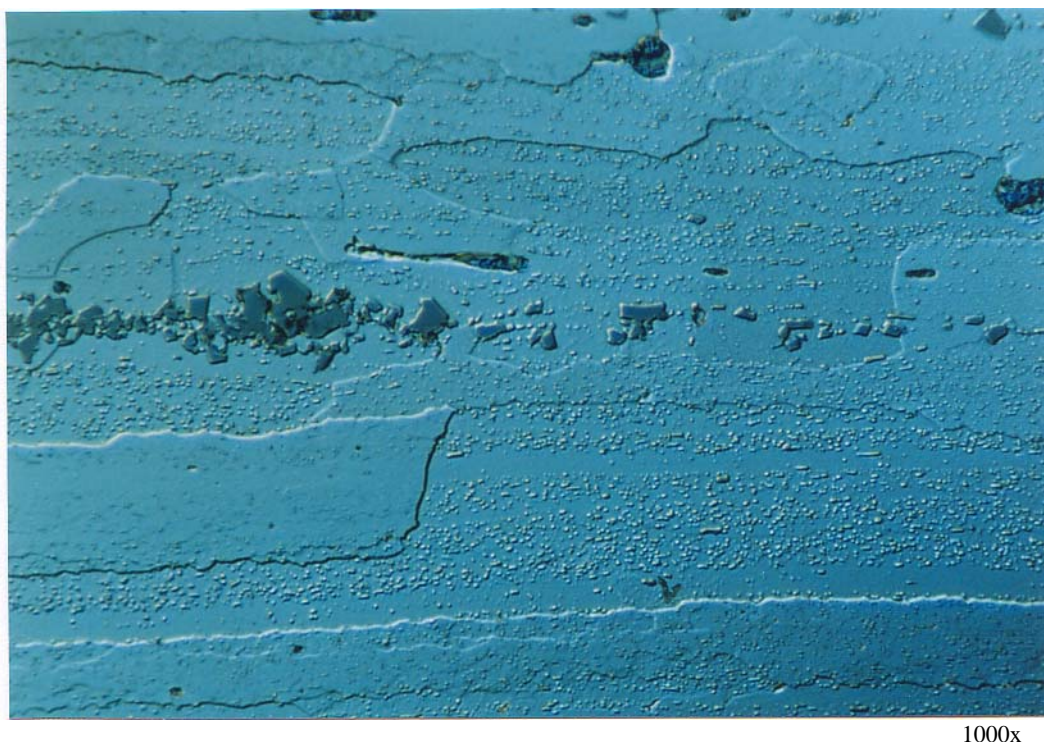


Figure 44: Texture of the Rolled Al Alloy AlMg4.5Mn/15G after Ion Beam Etching (Specimen 5), Micrograph with DIC, Grain Boundaries Developed, Grain Surfaces Rough to Grainy, Section from Figure 43, Parameter: 6kV / 405 μ A / 8mins, 55°, rotating,

3.1.4.6 AlMg4.5Mn/25G, Annealed and Rolled, Strained Texture (non-hardenable)

Specimen 6a	6 / 385 / 6, 55°, rotating	⇒ Grain boundaries etched, grain surfaces slightly rough to grainy (in the center more), inclusions or intermetallic phases o.k.
	+ 6 / 405 / 2, 55°, rotating	⇒ Etching better, grain surfaces in the center more rough to grainy
Specimen 1	6 / 405 / 8, 67.5°, rotating	⇒ Grain boundaries slightly etched, precipitates as well, intermetallic slanted, thus far o.k.
	+ 6 / 405 / 3.4, 65°, rotating	⇒ fine precipitates on grain surfaces, grain boundaries only developed weakly, phases o.k., etching so far o.k.
Specimen 2	6 / 405 / 12, 65°, rotating	⇒ Grain boundaries slightly etched, precipitates on grain surfaces
	+ 6 / 405 / 6, vertical	⇒ precipitates in the center of the etch mark at the grain boundaries (possible artifacts), artifacts also on grain surfaces (deformation) Etching for DIC good, for reflected light good up to 200x

Note:

This alloy has a similar etching response to the previous alloy. Here also there is the impact of the ion incident angle on the texture development.

3.1.4.7 Various Al Alloys

In order to examine and substantiate the dependence of the bombardment angle on the texture development, comprehensive etching tests were performed. To create the same pre-requisites for the 'angle dependence', a height of 10 mm with a diameter of 25 mm was used for the tests. Normally an acceleration voltage of 6 keV was run. The etching time was between 4 mins and 12 mins. The following alloys were examined:

AlMg3 (non-hardenable)
 AlMgSi (hardenable)
 AlZnMgCu1.5 (hardenable)
 AlCuMg1 / banded structure (hardenable)
 AlMg4.5Mn0.7 / banded structure (non-hardenable)
 Casting AlMgSi0.5 (hardenable)
 Casting AlMgSi1 (hardenable)
 Casting AlZnMgCu1.5 (hardenable)
 Casting AlZn4.5Mg1 (hardenable)

Independent of whether the alloy was hardenable or non-hardenable, the following tendencies could be ascertained:

The grain surfaces appear rough to grainy with low etching angles (about 55° to 65°).

The grain boundaries are normally etched at medium etching angles (about 30° to 50°)

The grain surfaces are similarly relatively smooth at medium angles (about 45°)

For steep angles (0° or vertical to 22.5°) and longer etching times, precipitates or strings of pearls can be formed on the grain boundaries. Similarly pimples can occur on the grain surfaces. All in all, a bombardment angle of about 45° appears to have proven to be best for good texture reproduction.

3.1.5 Mg and Mg Alloys

3.1.5.1 High-Purity Mg

Specimen 4A	6 / 405 / 16, 60°, rotating	⇒ cleaned and grain boundaries slightly etched
	+ 6 / 405 / 10, 55°, rotating	⇒ outer ring slightly etched
	+ 6.5 / 460 / >10, 55°, rotating	⇒ Super etching! Grain boundaries etched

3.1.5.2 Mg Casting Alloy AM50 (Figures 45 to 49)

Specimen 5	6 / 435 / 6, 60°, rotating	⇒ Specimen cleaned except for the center of the specimen, i.e. in the center less good, at the edge slight etching of the grains
	+ 6 / 445 / 6, 45°, rotating	⇒ Phases cleaned, also in the center of the specimen, grain surfaces slightly rough to

grainy, POL effect somewhat reinforced
 + 6 / 435 / 5.2, 10°, rotating ⇒ for POL very good, grain surfaces in the center very rough to grainy

Specimen A1 6 / 435 / 6, 65°, rotating ⇒ Specimen smoothed, gray phase also leveled,
 + 6 / 425 / 3.2, 45°, rotating ⇒ Specimen further smoothed
 + 6 / 407 / 1.4, 30°, rotating ⇒ Grain boundaries well developed
 + 6 / 435 / 2, 15°, rotating ⇒ for POL **Super etching!**

Note:

For Mg alloys, the ion beam is initially darker except for a light outer edge. A slight etching takes place. Thereafter the beam is lighter again, the consequence being a stronger etching attack. The grains are rough to grainy. a color effect is created when observing with polarized light,. If the grain surfaces are smooth after etching, the POL effect does not occur.

3.1.5.3 Mg Die Casting AM50

Specimen 1.4 6 / 435 / 2mins 60°+2.2mins 62°, ⇒ Specimen cleaned and smoothed rotating
 + 6 / 435 / 1.2, 40°, rotating ⇒ Grain boundaries slightly etched, weak POL effect
 + 6 / 435 / 1.4, 30°, rotating ⇒ further etching of the grain surfaces POL effect now better
 + 6 / 435 / 2, 15°, rotating ⇒ Center over-etched, grain surfaces very rough to grainy, however o.k.

Note:

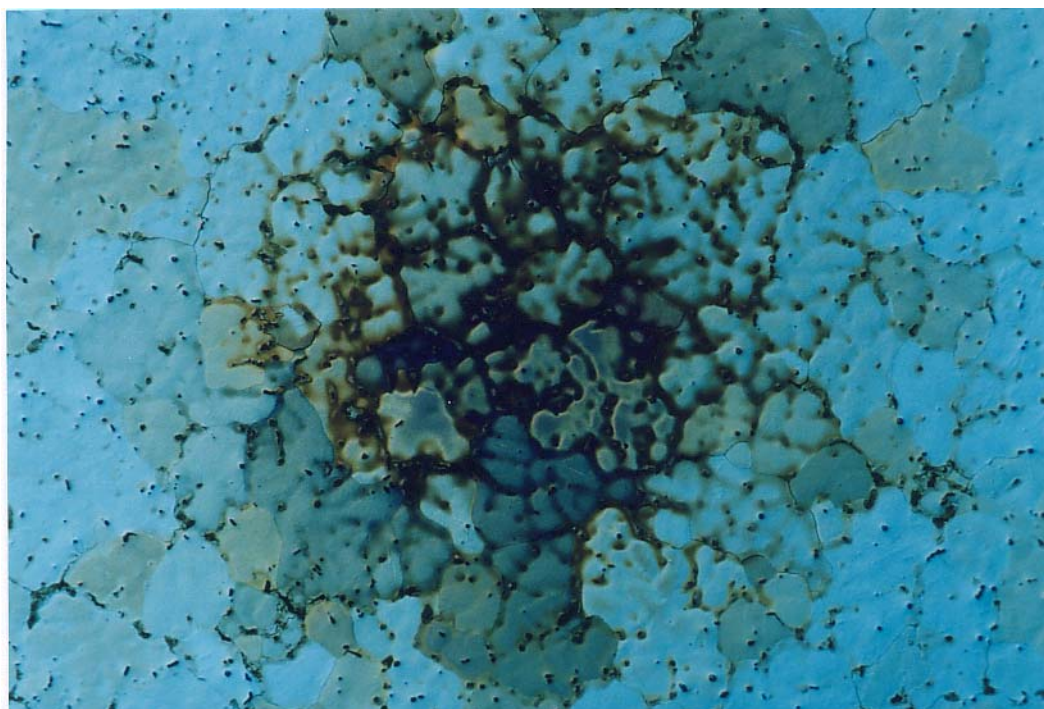
The etch response is similar to the previously listed casting alloy. The grains are much finer due to the procedure. They are easily identified in polarized light.

3.1.5.4 Mg Alloy AZ91 (Figures 50 to 52)

Specimen 2A 6 / 435 / 4.2, 65°, rotating ⇒ Specimen smoothed, phases slightly etched
 + 6 / 435 / 4.2, 65°, rotating ⇒ Etching in POL already quite good but insufficient
 + 6 / 435 / 1.4, 37.5°, rotating ⇒ now in POL much better
 + 6 / 435 / 1, 37,5°, rotating ⇒ in POL still somewhat more high-contrast, leave it like that!

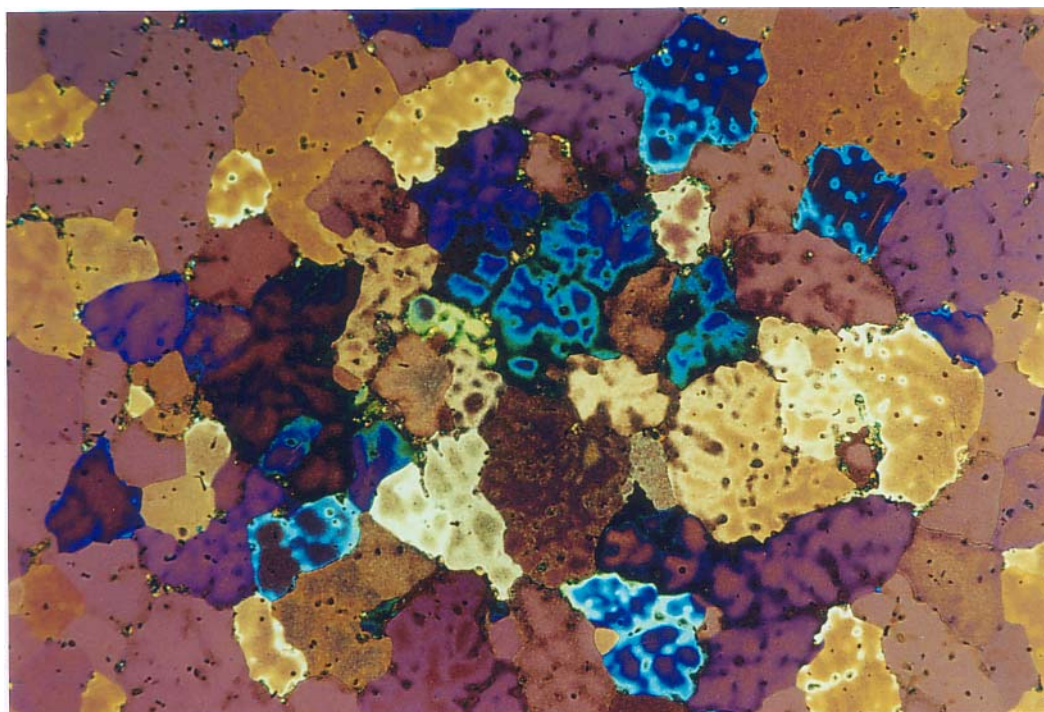
Note:

With this alloy also a POL effect is first achieved when the grain surfaces are adequately rough to grainy.



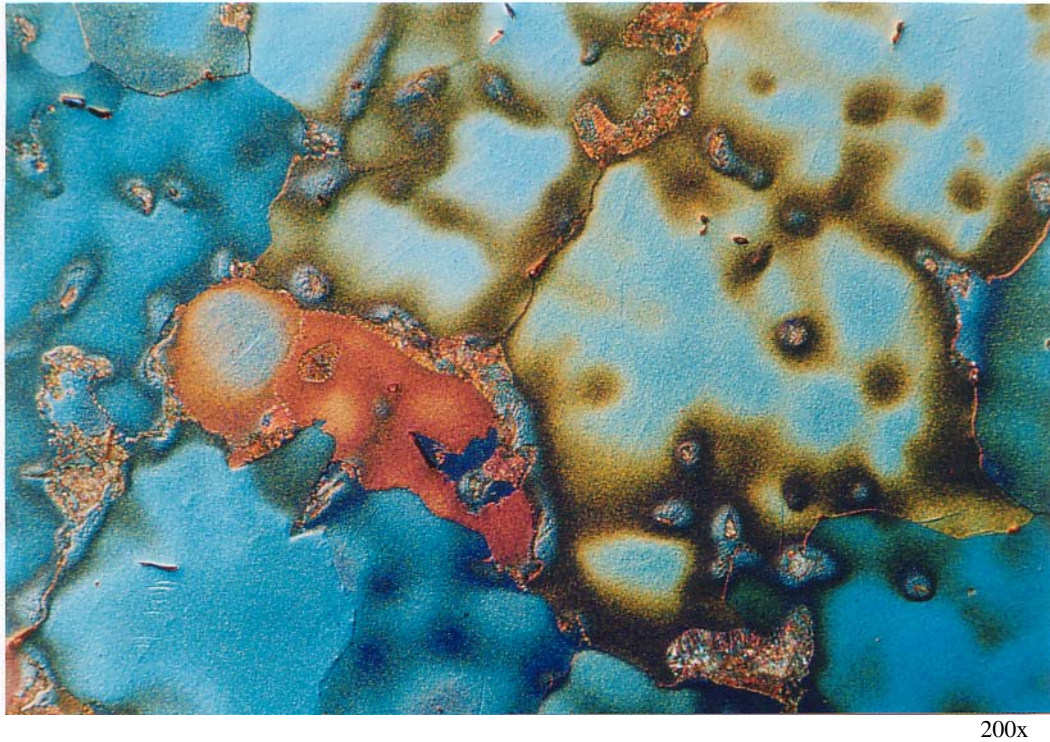
50x

Figure 45: Etch mark for the Mg Casting Alloy AM50 after ion bombardment (Specimen 5 /6/), Micrograph with Differential Interference Contrast (DIC) in the optical beam path, Parameter: 6kV / 435 μ A / 6mins, 60°, rotating + 6kV / 445 μ A / 6mins, 45°, rotating + 6kV / 435 μ A / 5.2mins, 10°, rotating



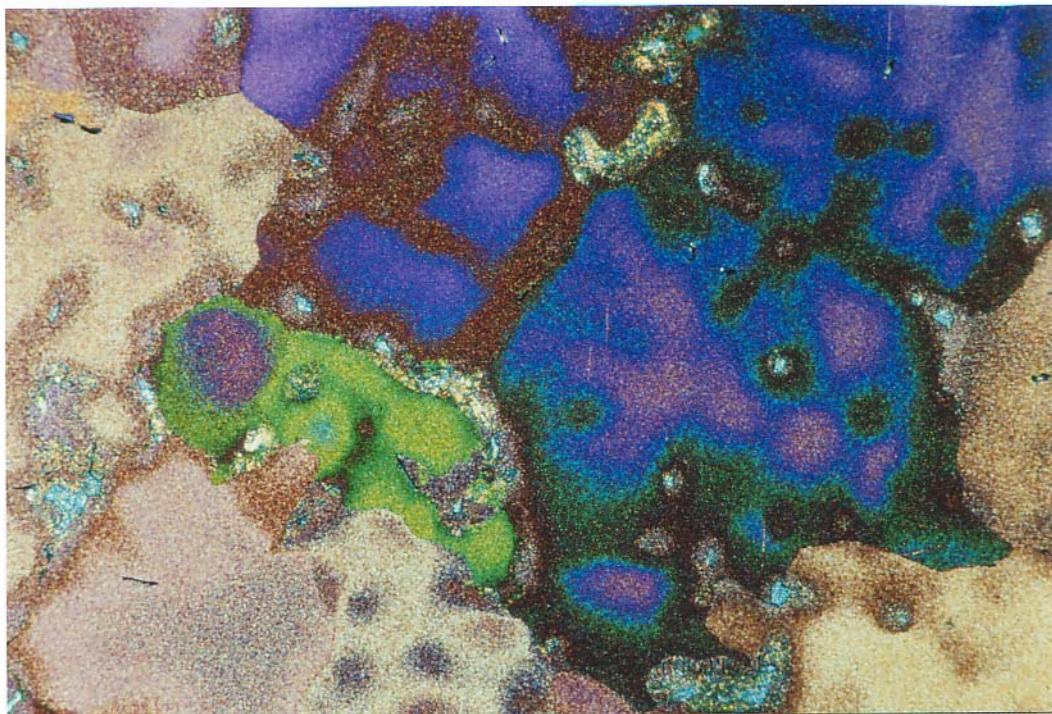
50x

Figure 46: Etch mark for of the Mg Casting Alloy AM50 after ion bombardment (Specimen 5 /6/), Micrograph as Figure 45, however with polarized beam path, grains retain depending on their orientation different color effects, segregation in the cast grains are visible (specimen somewhat tilted)



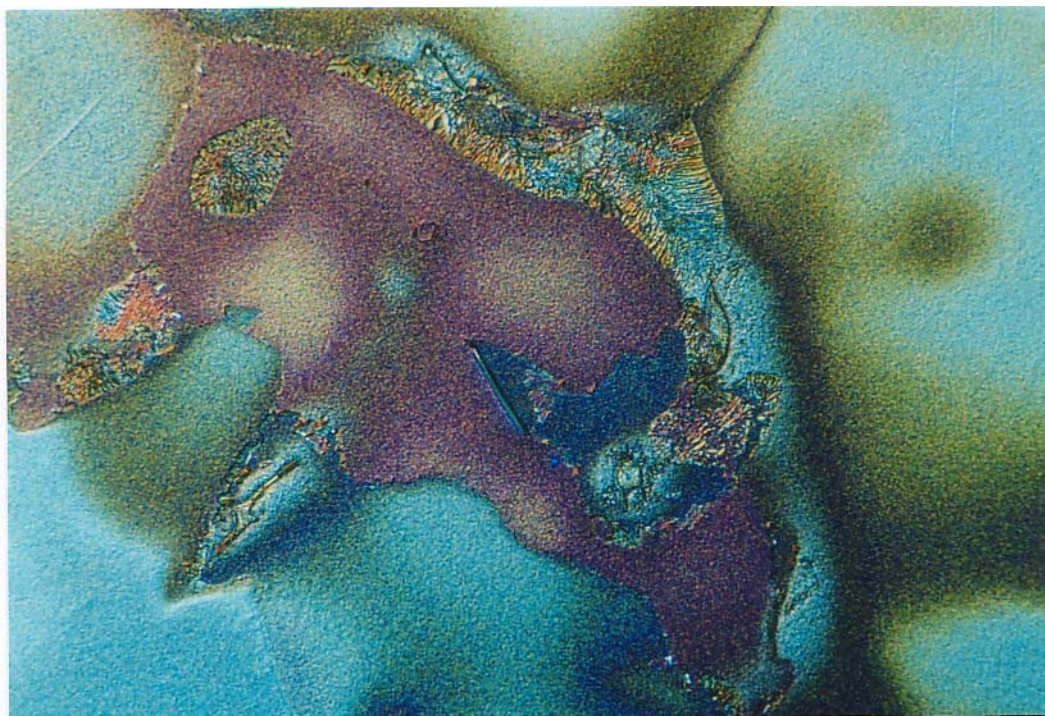
200x

Figure 47: Texture of the Mg Casting Alloy AM50 after Ion Beam Etching (Specimen 5 /6/), Micrograph with DIC, here again the grains are, depending on their orientation different colors, segregations are similarly visible, Enlarged Sectional Micrograph of Figure 45 (Specimen somewhat tilted)



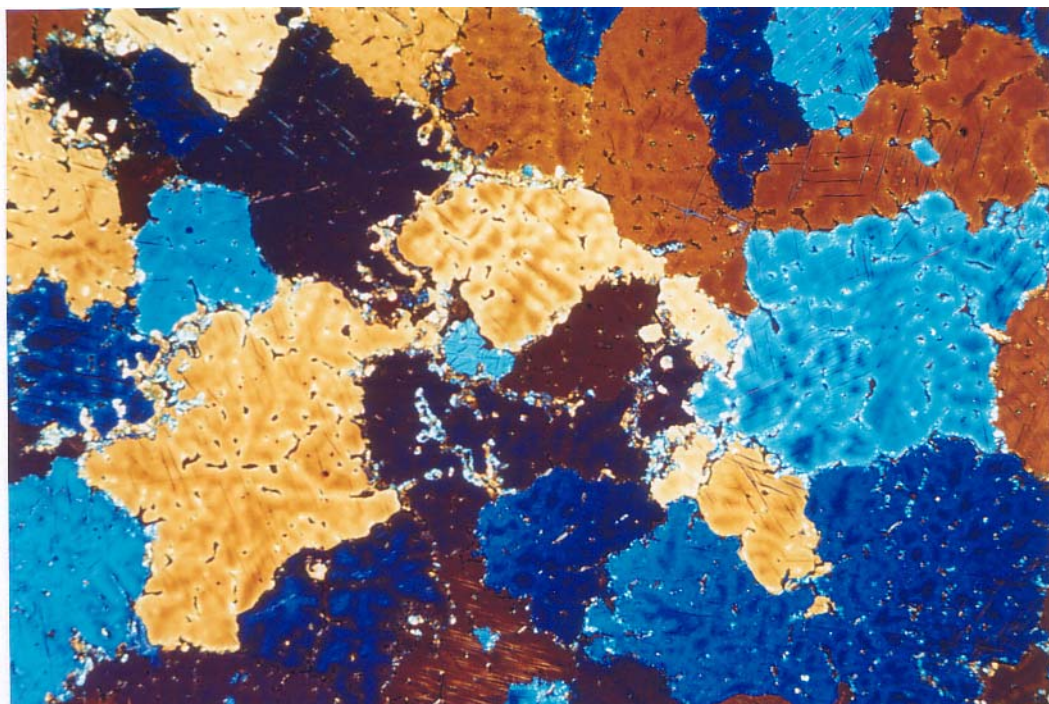
200x

Figure 48: Texture of the Mg Casting Alloy AM50 after Ion Beam Etching (Specimens 5 /6/), Micrograph as Figure 47, however with POL-filters, segregations and Different grain orientation visible, Enlarged Section of Figure 46



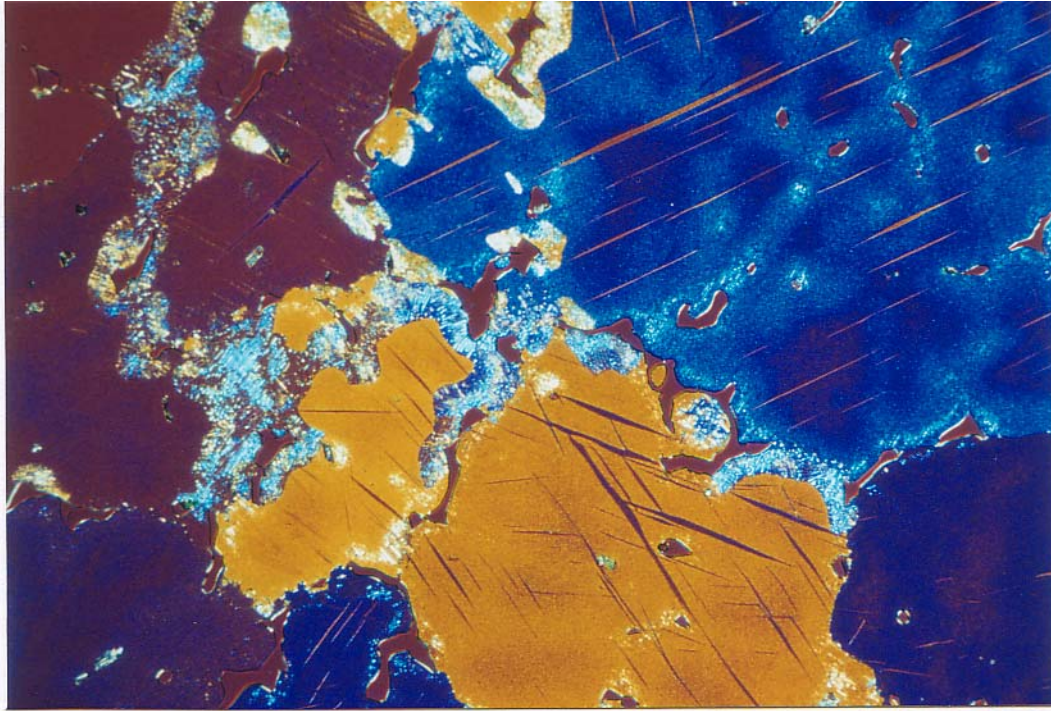
500x

Figure 49: Texture of the Mg Casting Alloy AM50 after Ion Beam Etching (Specimens 5 /6/), Micrograph with DIC, Enlarged Section of Figure 47, Surface rough to grainy



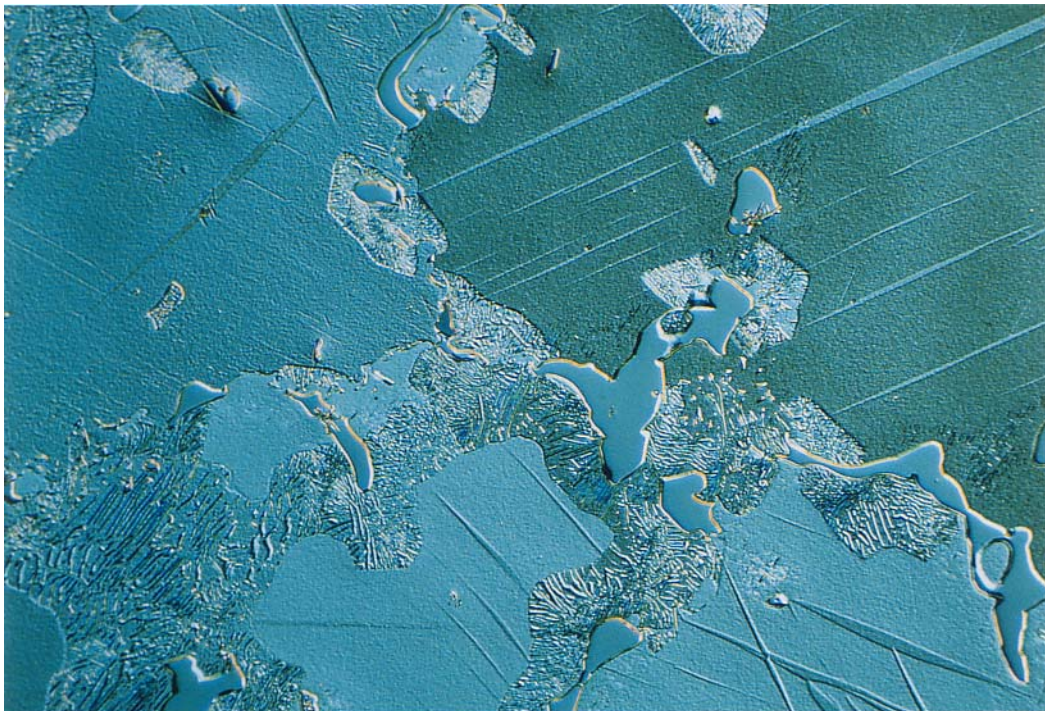
50x

Figure 50: Texture of the Mg Casting Alloy AZ91 after Ion Beam Etching (Specimen 2A), Micrograph with POL filters, grains retain depending on their orientation different color effects, Segregations in the casting grains are identifiable
 Parameter: 6kV / 435 μ A / in total 8.4mins, 65°, rotating
 + 6kV / 435 μ A / in total 2.4mins, 37.5°, rotating



200x

Figure 51: Texture of the Mg Casting Alloy AZ91 after Ion Beam Etching (Specimen 2A), Micrograph with POL filters, grains retain depending on their orientation different color effects, Segregations in the casting grains are identifiable, Sectional Enlargement of Figure 50



500x

Figure 52: Texture of the Mg Casting Alloy AZ91 after Ion Beam Etching (Specimen 2A), Micrograph with DIC, intermetallic phase Mg_3Al and perlite textures etched at the grain boundaries, Parameter: cf. Figure 50, multiple etching

3.1.6 Superconductors

3.1.6.1 Superconductor Cu+NbTi

Specimen 2 (longitudinal)	6 / 405 / 10, 45°, rotating	⇒ Cu for DIC adequate, NbTi fibers not etched, however diffusion zone visible at the border area
	+ 6 / 405 / 1.2, vertical, rotating	⇒ Etching for DIC and reflected light o.k. thus far, for NbTi fibers only diffusion zone visible
Specimen 3 (lateral)	6 / 405 / 12, 45°, rotating	⇒ Cu for DIC good, diffusion zone for NbTi fibers
	+ 6 / 400 / 3.2, vertical, rotating	⇒ Cu more strongly etched, diffusion zone for NbTi fibers more strongly, in NbTi fine texture, etching good thus far

3.1.6.2 Superconductor CuSn+NbTi+Ta with Cu Layer (Figures 53 to 57)

Specimen 2 (longitudinal)	6 / 405 / 12, 45°, rotating	⇒ CuSn and Cu for DIC very good, NbTi fibers and Ta coating not etched, however diffusion zone visible, center of the etch mark hardly etched
	+ 6 / 405 / 2.4, vertical, rotating	⇒ CuSn and Cu very good, in NbTi fibers and Ta coating a texture is indicated
	+ 6 / 390 / 2, 10°, rotating	⇒ NbTi fibers and Ta coating slightly etched, CuSn and Cu somewhat more strongly attacked, o.k. thus far.
Specimen 5 (lateral)	6 / 365 / 8, 45°, rotating	⇒ NbTi fibers and Ta coating slightly etched, CuSn and Cu in part somewhat more strongly etched, for DIC o.k.



Figure 53: Superconductor CuSn+NbTi+Ta with Cu Layer after Ion Beam Etching (Specimen 2, longitudinal), Overview Micrograph with DIC, Parameter: multiple etching, please see above.



Figure 54: Superconductor CuSn+NbTi+Ta with Cu Layer after Ion Beam Etching (Specimen 2, longitudinal), Ta with Cu Coating, Section from Upper Area of Figure 53, Micrograph with DIC,
 Parameter: 6 kV / 405 μ A / 12 mins, 45°, rotating
 + 6 kV / 405 μ A / 2.4 mins, vertical, rotating + 6 kV / 390 μ A / 2 mins, 10°, rotating



Figure 55: Superconductor CuSn+NbTi+Ta with Cu Layer after Ion Beam Etching (Specimen 2, longitudinal), CuSn with Ta-Coating, Section from Upper Area of Figure 53
Micrograph with DIC, Parameter: multiple etching, cf. Figure 53



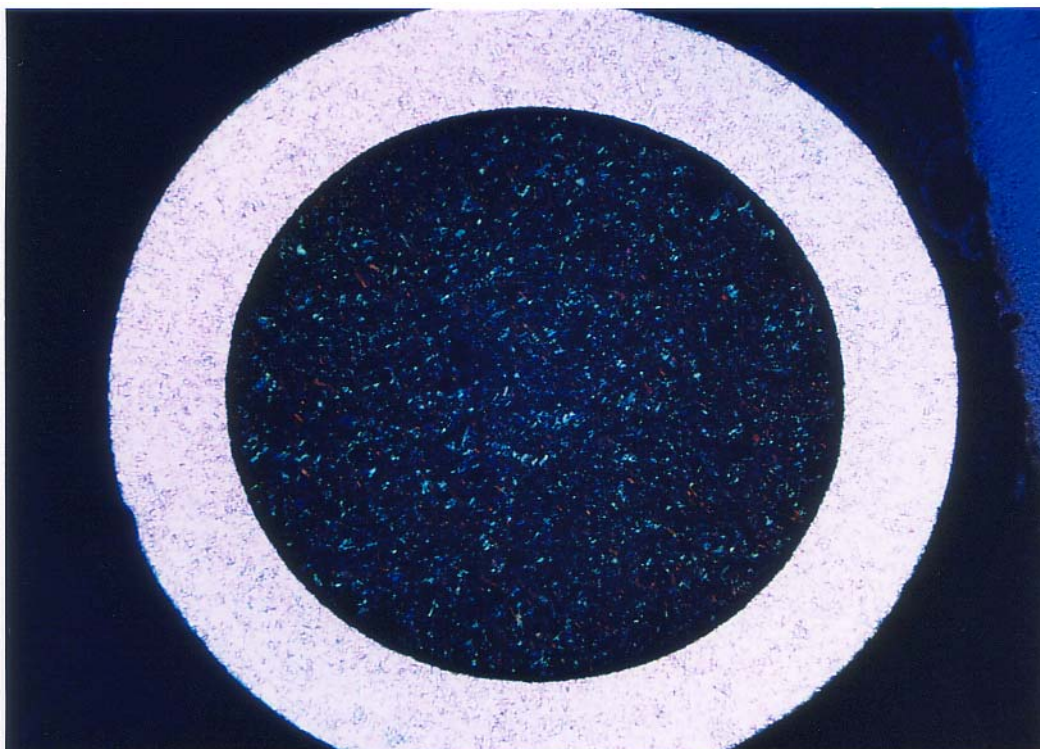
Figure 56: Superconductor CuSn+NbTi+Ta with Cu Layer after Ion Beam Etching (Specimen 2, longitudinal), CuSn Matrix with NbTi fibers, Lower Area of Figure 53, Micrograph with DIC, Parameter: 6 kV / 405 μ A / 12 mins, 45°, rotating
+ 6 kV / 405 μ A / 2.4 mins, vertical, rotating
+ 6 kV / 390 μ A / 2 mins, 10°, rotating

(Figures 58 to 62)

Specimen 1 6 / 385 / 4.4, 45°, rotating ⇒ Ag grain surface etched, ceramic core textured, POL effect.

Note:

The grain boundaries and twins of the mantles of AgMg and Ag are well developed for DIC imaging. Similarly the ceramic core of PbBeSr/CaCuO reveals a fine texture. By sputtering with AuPd, the contrast of the ceramic is enhanced and the AgMg mantles are preserved against rapid oxidization. For the ceramic (composition unknown) in an Ag mantle, ion beam etching leads to a POL effect.



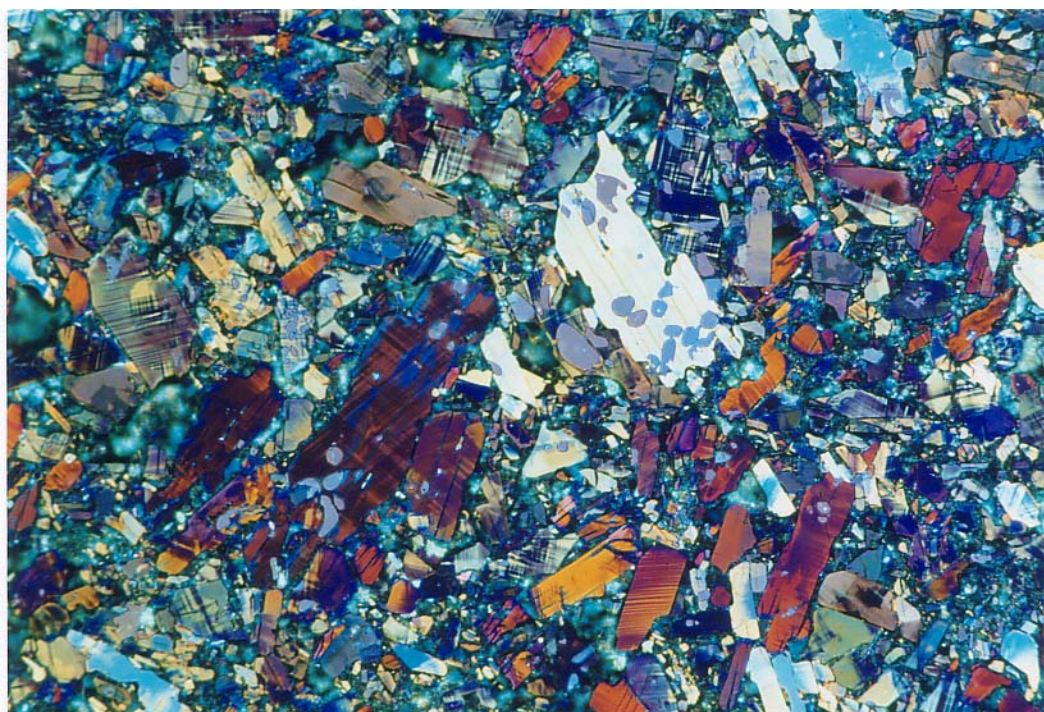
12.5x

Figure 58: Overview of the Superconductor with Ag Mantle and Ceramic Core, Ion Beam Etched (Specimen 1), Micrograph with POL Filters, Parameter: 6 kV / 385 μ A / 4.4 mins, 45°, rotating



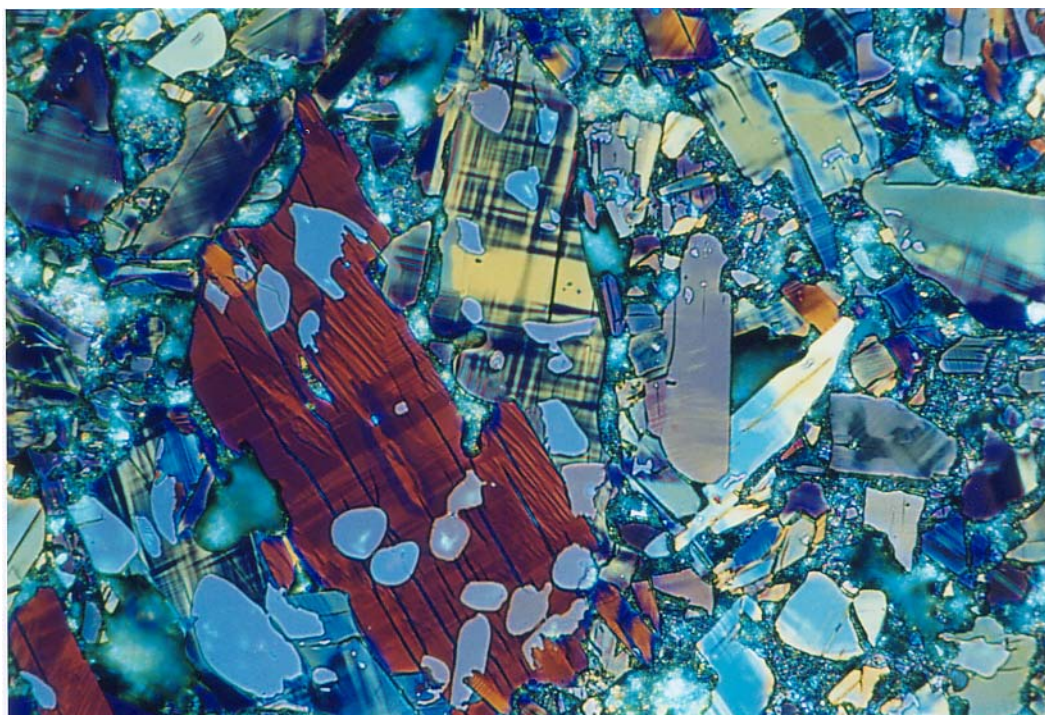
50x

Figure 59: Partial Area of the Superconductor of Ag Mantle with Ceramic Core after Ion Bombardment (Specimen 1), Grain Boundaries and Twins of the Ag Mantle Developed, Micrograph with POL Filters, Parameter: 6 kV / 385 μ A / 4.4 mins, 45°, rotating



200x

Figure 60: Ceramic Core of the Superconductor with Ag Mantle after Ion Beam Etching (Specimen 1), Ceramic Core Components Reveal Differing Color Effects Micrograph with POL Filters, Parameter: 6 kV / 385 μ A / 4.4 mins, 45°, rotating



500x

Figure 61: Ceramic Core of the Superconductor with Ag Mantle after Ion Beam Etching (Specimen 1), Ceramic Core Components Reveal Differing Color Effects Micrograph with POL Filters, Enlarged Section of Figure 60



500x

Figure 62: Ceramic Core of the Superconductor with Ag Mantel after Ion Beam Etching (Specimen 1), same position as Figure 61, Micrograph however with Differential Interference Contrast, Parameter: 6 kV / 385 μ A / 4.4 mins, 45°, rotating

3.1.7 Permanent Magnets, Composition: FeNdB(1%), hard-magnetic

Small old Specimen 4 (Figures 63 to 65)

- Etch gas Ar** 6.5 / 480 / 5, 40°, rotating ⇒ Grain boundary not yet etched, phases already edged
- + 6.5 / 515 / 4.4, 10°, rotating ⇒ Center of specimen somewhat over-etched (for reflected light o.k.), Grain boundary in part continuous, in part etched point-like colored phase textured
- + 6.5 / 505 / 1.2, 10°, rotating ⇒ Etching (particularly edge layer) altogether better
- + 6.5 / 495 / 1.4, 10°, rotating ⇒ Etching (particularly edge layer) still better
- + 6.5 / 515 / 1.0, 10°, rotating ⇒ Etching thus far o.k.
- Etch gas O₂** + 8 / 445 / 4, vertical, rotating ⇒ brownish tarnished etching, grains colored equally, by contrast phases in the material and edge layers of Ni and Cu tinted differently → o.k.

Large old Specimen 5

- Etch gas Ar** 6.5 / 505 / 5.2, 30°, rotating ⇒ Grain boundary already etched, however not completely
- + 6.5 / 495 / 2.4, 30°, rotating ⇒ Etching (particularly edge layer) somewhat better
- + 6.5 / 490 / 2.4, 22.5°, rotating ⇒ Etching further improved
- + 6.5 / 490 / 2.12, 15°, rotating ⇒ Grain boundary not yet quite complete, phases in part strongly over-etched, otherwise o.k.
- + 6.5 / 465 / 1.4, 10°, rotating ⇒ Center over-etched and rough to grainy, only partially linked grain boundary

Note:

It is important for the permanent magnets that the etchings and micrographs are made immediately after preparation, as this material corrodes very quickly and the structure is thus falsified. To increase contrasting between the different phases and the protective layers of Ni and Cu, it is appropriate to treat the material with oxygen as a reaction gas after the Ar etching.

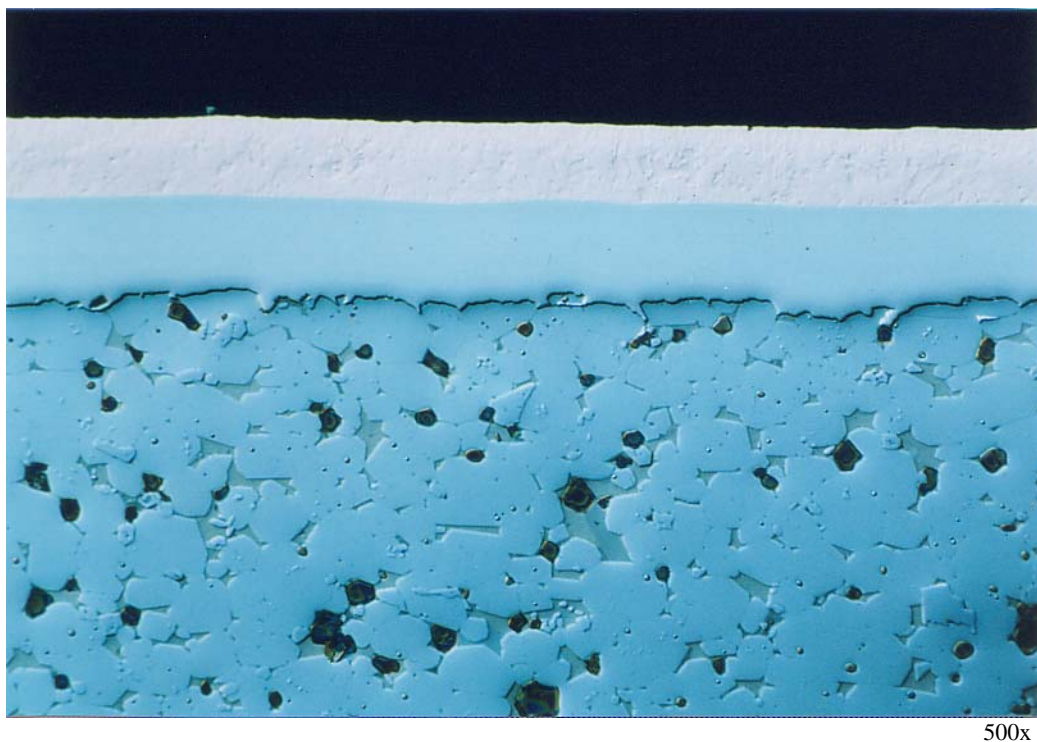


Figure 63: Structure from the Edge of the Permanent Magnet FeNdB(1%), unetched (small old Specimen 4), Micrograph with DIC, Edge Layers of Cu and Ni

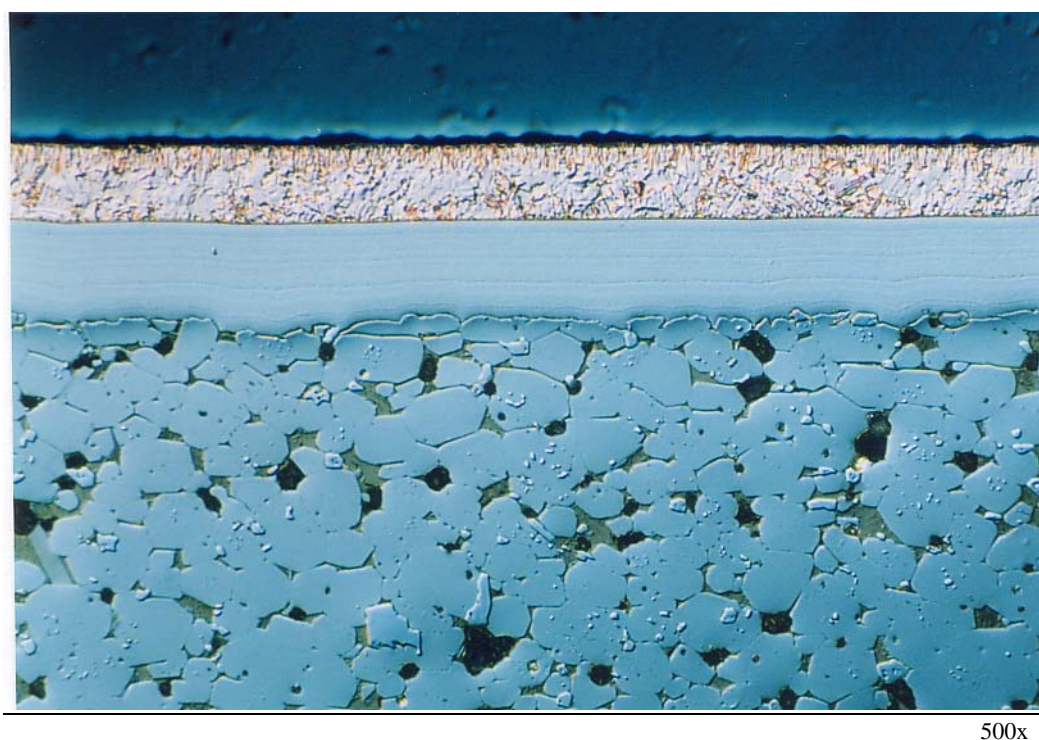
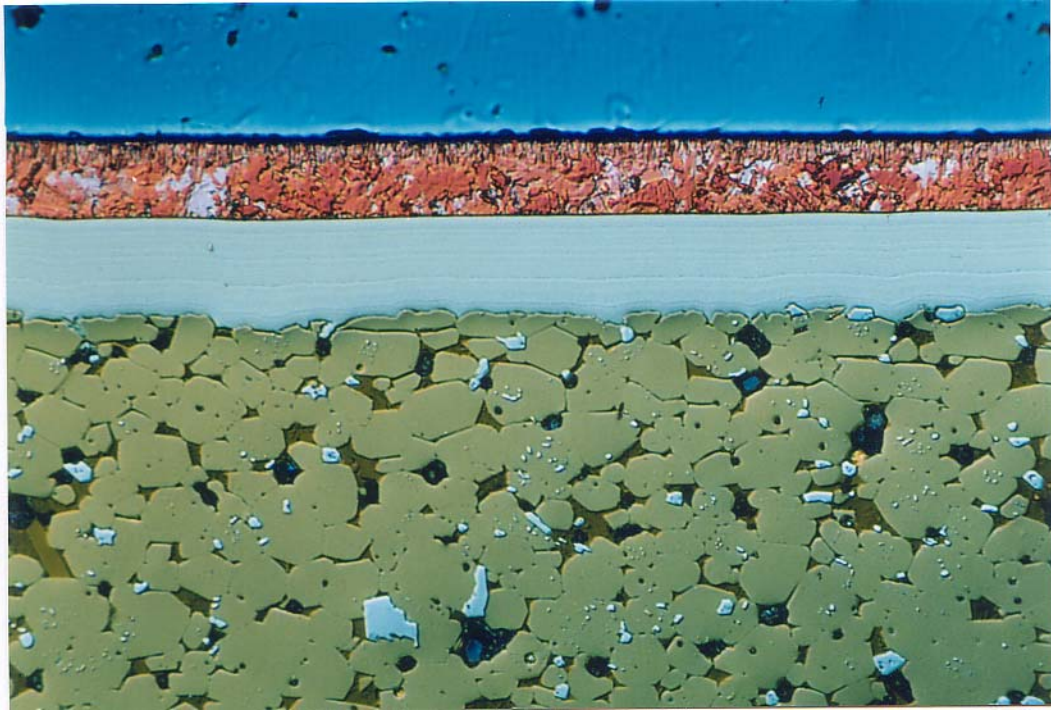


Figure 64: Structure from the Edge of the Permanent Magnet FeNdB(1%) after Ion Beam Etching (small old Specimen 4), Micrograph with DIC, practically the same position as Figure 63, Texturing of Matrix, Cu and Ni Layers, Parameter: multiple etching, please see above.



500x

Figure 65: Structure from the Edge of the Permanent Magnet FeNdB(1%) after Ion Beam Etching with Ar and additionally with O₂ (small old Specimen 4), Micrograph with DIC, in the same position as Figure 64, improved contrast by means of tarnished layer, Parameter: multiple etching.

3.1.8 Layered Systems

3.1.8.1 Build-up Welds (Ni alloy on boilerplate HII)

Small specimen 6 / 405 / 8, vertical ⇒ for DIC already too strong, good for reflected light → new

Small specimen (newly prepared)

Etch gas Ar 6 / 455 / 8, 45°, rotating ⇒ Etching already quite good, mark larger than before

+ 6 / 470 / 4.4, 45°, rotating ⇒ Etching for DIC thus far **o.k.**

Large specimen (Figures 66 and 67)

Etch gas Ar 6 / 466 / 8, 45°, rotating ⇒ Etching for DIC already very good

+ 6 / 450 / 1.2, 45°, rotating ⇒ Grain boundary etched somewhat stronger but also fine pores

Etch gas O₂ + 8.5 / 385 / 5.2, vertical, rotating (*Specimen off center*) ⇒ Tarnished etching o.k.

3.1.8.2 Build-up Welds (Ni alloy on boilerplate HII), Triple Layer, Rolled

Specimen 1

Etch gas Ar 6 / 405 / 8, vertical ⇒ Center somewhat fiercely etched, for reflected light o.k., edge for DIC o.k.

Specimen 4 (Specimen for microprobe investigation)

Etch gas Ar 6 / 435 / 8, 45°, rotating ⇒ already slight etching of layer and Base material

+ 6 / 435 / 4.4, 45°, rotating ⇒ Grain boundary in layer and Base material better developed → super

Etch gas O₂ + 9.5 / 500. / 3.2, vertical ⇒ Etching more contrasting

+ 8 / 400. / 2, vertical, rotating ⇒ *Specimen not taken out of the chamber*

+ 9.5 / 500. / 4, vertical, rotating ⇒ Super tarnish etching

+ 9.5 / 500. / 4, vertical, rotating ⇒ even better for DIC and reflected light. o.k.

Note:

The chemical characteristics between the build-up welds of an Ni alloy and the substrate of plain steel reveal enormous differences so that simultaneous structure development of both compound partners is not possible with chemical or electrochemical etch agents. With ion beam etching, simultaneous structuring is achieved as the sputtering yields are similar. In addition, the contrast can be enhanced further if, after etching with argon, a tarnish etching with oxygen is performed. The grains in the weld are tinted in accordance with their orientation.

3.1.8.3 Build-up Welds (Ni Alloy on GGG40)

Specimen 1

<i>Etch gas Ar</i>	6 / 405 / 12, 52.5, rotating	⇒ for DIC very good (perlite in base material not etched)
<i>Etch gas O₂</i>	+ 8.5 / 240 / 4, vertical, rotating (<i>Specimen off center</i>)	⇒ Tarnish etching good thus far
	+ 8.5 / 385 / 2.2, vertical, rotating (<i>Specimen off center</i>)	⇒ Etching more contrasting

Note:

While the weld in this compound is textured by ion etching, the martensite and perlite structure in the heat impact zone and in the base material are not attacked. In order to develop this structure in similar fashion, etching with, for example, 2 % HNO₃ in alcohol can be performed before or after ion beam etching. A further enhancement of contrast of the weld can, as has been mentioned several times, be achieved by bombarding with O₂



100x

Figure 66: Build-Up Welds derived from a Ni Alloy on Boilerplate HII after Bombardment with Ar Ions (large specimen), Micrograph with DIC, Substrate and Welds Etched, Parameter: 6 kV / 466 μ A / 8 mins, 45°, rotating + 6 kV / 450 μ A / 1.2 mins, 45°, rotating



100x

Figure 67: Build-Up Welds derived from a Ni Alloy on Boilerplate HII after Bombardment with Ar-Ions and additionally with Oxygen (large specimen), Micrograph with DIC, Same position as Figure 66, enhanced contrast by means of Tarnish Layer, Parameter: Ar Etching (please see above) + O₂ Etching (8.5 kV / 385 μA / 5.2 mins, vertical, rotating)

3.1.8.4 Welds (Hastalloy) with Ni Intermediate Layer on Plain Steel

Specimen 1 (Figures 68 to 72)

Etch gas Ar 6 / 405 / 16, 35°, rotating ⇒ Grain boundary slightly etched, Ni layer good for DIC
 + 6 / 405 / 6, 10°, rotating ⇒ Etching very good, leave it like that, o.k.
Etch gas O₂ + 9.5 / 480 / 4, vertical, rotating ⇒ Contrast better, particularly of the Ni layer

Specimen 6

Etch gas Ar 6.5 / 435 / 16, 22.5°, rotating ⇒ already quite good, for DIC almost suitable
 + 6.5 / 435 / 8, 22.5, rotating ⇒ Etching in total better
 + 6.5 / 435 / 8.2, 22.5°, rotating ⇒ Etching seen overall even better
 + 6.5 / 445 / 7.2, 22.5°, rotating ⇒ Welds, layer and base material thus far o.k.
Etch gas O₂ + 8.5 / 430 / 4, vertical, rotating ⇒ Contrast better as a result of tarnish etching

Note:

The etch impact for welds of Hastalloy is weak, compared to the Ni intermediate layer and to the base material of plain steel. Therefore contrast enhancement as a result of a tarnish etching using O₂ as the reaction gas also proves to be advantageous here.



100x

Figure 68: Overview of the Weld Link of Hastalloy on Plain Steel with Ni Intermediate Layer after Bombardment with Ar Ions (Specimen 1), Micrograph with DIC, Slight Texturing of Matrix, Welds and NI Layer,

Parameter: 6 kV / 405 μ A / 16 mins, 35°, rotating + 6 kV / 405 μ A / 6 mins, 10°, rotating



100x

Figure 69: Overview of the Weld Link of Hastalloy on Plain Steel with Ni Intermediate Layer after Bombardment with Ar Ions and additionally with Oxygen

(Specimen 1), Same Position as Figure 68, Enhanced Contrast by means of Tarnish Layer, Parameter: Ar Etching (please see above) + O₂ Etching (9.5 kV / 480 μ A / 4 mins, vertical, rotating)



200x

Figure 70: Weld Link of Hastalloy on Plain Steel with Ni Intermediate Layer after Bombardment with Ar Ions and additionally with Oxygen (Specimen 1), Enlarged Section of Figure 69, Enhanced Contrast by means of Tarnish Layer



500x

Figure 71: Weld Link of Hastalloy on Plain Steel with Ni Intermediate Layer after Bombardment with Ar Ions (Specimen 1), Micrograph with DIC, Texturing of Matrix, Welds and Ni Layer, Enlarged Section of Figure 68

Parameter: 6 kV / 405 μ A / 16 mins, 35°, rotating + 6 kV / 405 μ A / 6 mins, 10°, rotating



500x

Figure 72: Weld Link of Hastalloy on Plain Steel with Ni Intermediate Layer after Bombardment with Ar Ions and additionally with Oxygen (Specimen 1), Same Position as Figure 71, Enhanced Contrast as a result of Tarnish Layer

3.1.8.5 PM Steel with Antiabrasion Layer (austenitic)

<u>Specimen 1</u>	6 / 405 / 16, 35°, rotating	⇒ Grain boundary in welds slightly etched, carbides similarly
	+ 6 / 405 / 6, 10°, rotating	⇒ Etching for DIC <u>very good</u>
<u>Specimen 4</u> (Specimen very high, 14mm)		
<i>Etch gas Ar</i>	6 / 405 / 16, 30°, rotating	⇒ Grain boundary in welds indicated, carbides slightly etched
	+ 6 / 405 / 6, 10°, rotating	⇒ Etching for DIC <u>very good</u>
<i>Etch gas O₂</i>	+ 9.5 / 490 / 4, vertical, rotating	⇒ slight contrasting as a result of tarnish etching

Note:

There are no chemical nor electrochemical agents for this compound which cause simultaneous structure development. Only as a result of ion beam etching could both compound partners be etched simultaneously.

3.1.8.6 Nickel Solder: Nimonic / NB30 (16h/710°C) (coarse phase)

Specimen 8F (not hardened) (Figures 73 to 75)

Etch gas Ar 6 / 440 / 4.2, 45°, rotating	⇒ Grain boundary and twin etched in Base material, phases (or edges of phases) similarly in solder
+ 6 / 435 / 1.4, 45°, rotating	⇒ Etching better but still too short
+ 6 / 435 / 1.2, 45°, rotating	⇒ Contrast now better
+ 6 / 435 / 1.2, 45°, rotating	⇒ Contrast enhanced further, etching o.k. base material
Etch gas O₂ + 8.5 / 445 / 3.4, vertical, rotating (<i>Specimen off center</i>)	⇒ Super tarnish etching

3.1.8.7 Nickel Solder: Nimonic / NB125 (fine phase)Specimen 13 (Figures 76 to 80)

Etch gas Ar 6 / 440 / 6.2, 45°, rotating	⇒ Grain boundary and twin well etched in Base material, phases (or edges of phases) similarly in solder
+ 6 / 425 / 2, 45°, rotating	⇒ Contrast enhanced, etching o.k.
Etch gas O₂ + 8.5 / 445 / 3.4, vertical, rotating (<i>Specimen off center</i>)	⇒ Super tarnish etching

3.1.8.8 Nickel Solder: Steel HFX / NB30 (coarse phase)Specimen 19

Etch gas Ar 6 / 435 / 4.2, 45°, rotating	⇒ Ni Martensite in Base material for DIC well etched, phases etched in solder, boundary line well visible
+ 6 / 440 / 2, 45°, rotating	⇒ Etching more contrasting
+ 6 / 425 / 1.4, 45°, rotating	⇒ Contrast even better → leave it like that
Etch gas O₂ + 8.5 / 435 / 4.4, vertical, rotating (<i>Specimen off center</i>)	⇒ even tarnish etching, Phases in solder etched further, grain surfaces in the solder matrix differently colored, segregations at transitions Solder /Base material visible, Ni martensite more contrasting

Note:

Grain boundaries, twins and also carbides in the Base material could be clearly developed by ion beam etching. The transition between solder and matrix and the different phases in the solder were considerably better contrasted by an additional tarnish etching with oxygen as a reaction gas.



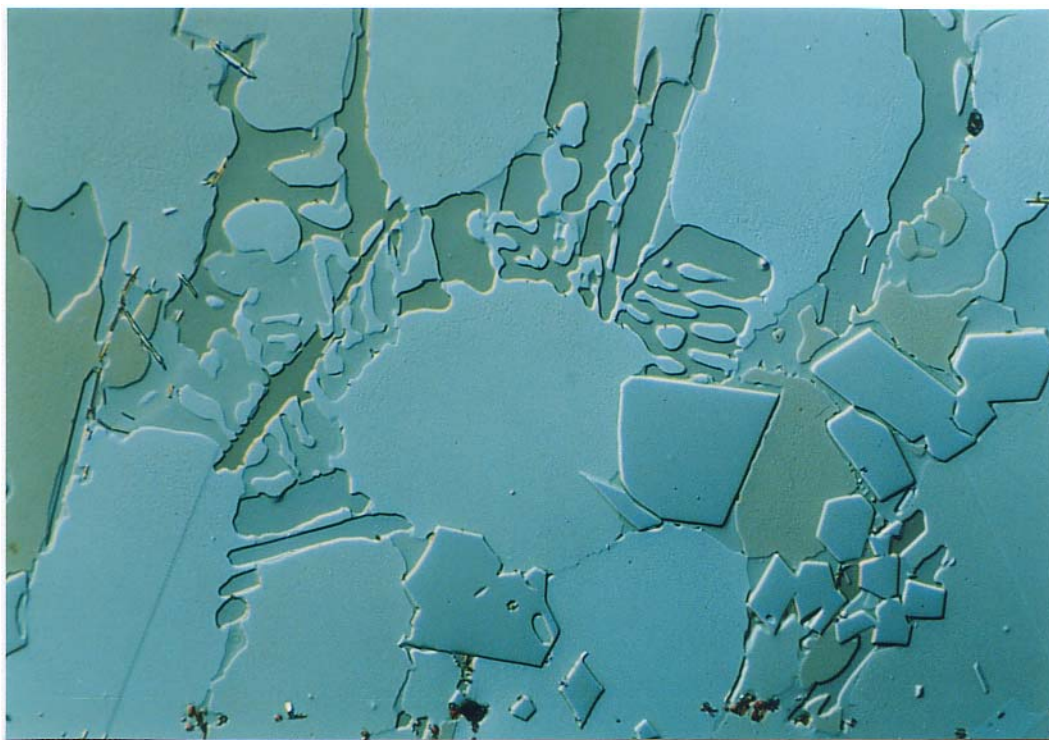
200x

Figure 73: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB30 (solder) after bombardment with Ar ions (Specimen 8F), Micrograph with DIC, Slight Texturing of Base Material, Solder and Transition, Parameter: 6 kV / \approx 435 μ A / in total 8.4 mins, 45°, rotating



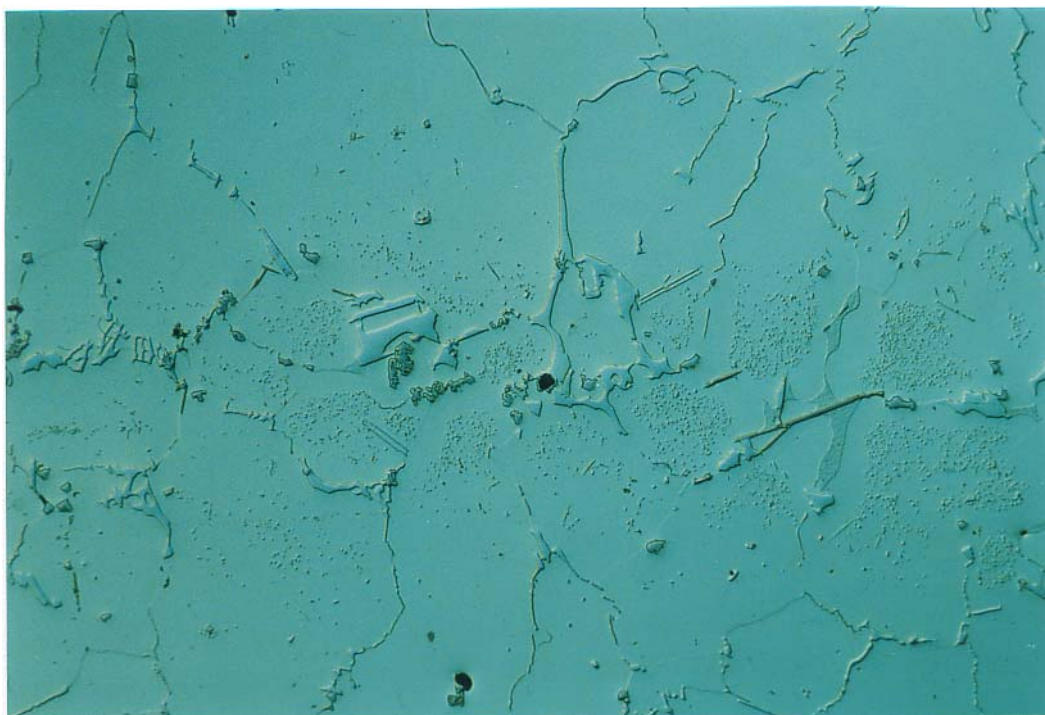
200x

Figure 74: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB30 (solder) after Bombardment with Ar ions and additionally with Oxygen (Specimen 8F), same position as Figure 73, Enhanced contrast as a result of the tarnish layer, Parameter: Ar Etching (please see above) + O₂ Etching (8.5 kV / 445 μ A / 3.4 mins, vertical, rotating)



500x

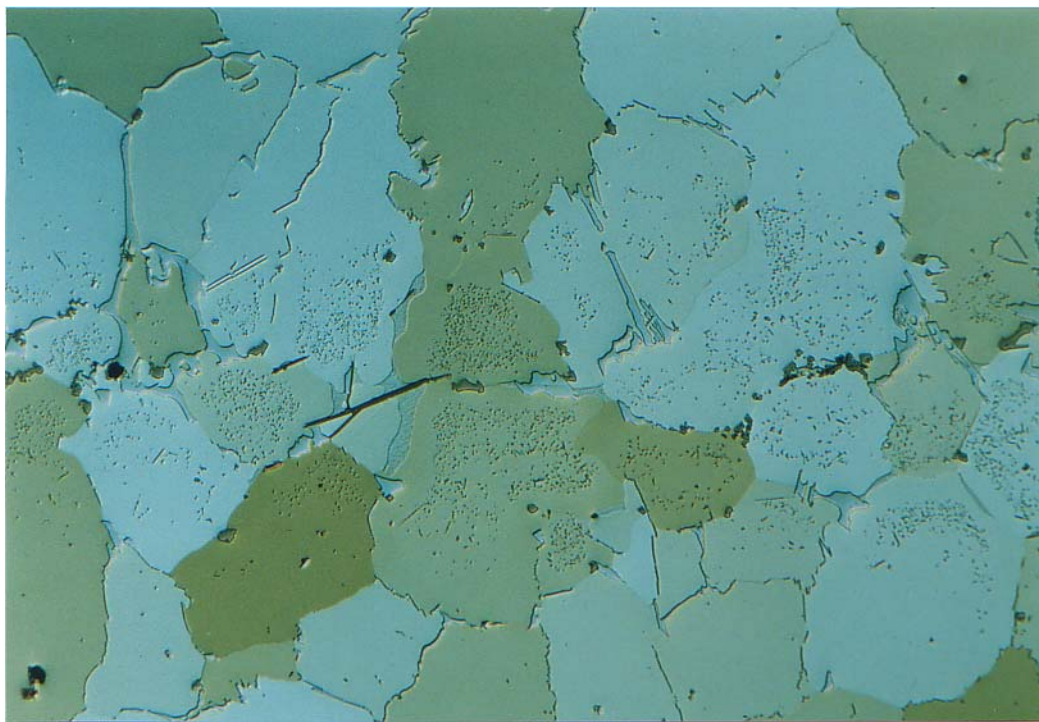
Figure 75: aged Joint made of the Nickel Alloys Nimonic (base material) and NB30 (solder) after Bombardment with Ar Ions and additionally with Oxygen (Specimen 8F), Section of Figure 74, Enhanced Contrast as a result of the Tarnish Layer, Parameter: cf. Figure 73



200x

Figure 76: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB125 (solder) after Bombardment with Ar Ions (Specimen 13), Micrograph with DIC, Slight Texturing of Base Material, Solder Matrix and Solder Phases,

Parameter: 6 kV / 440 μ A / 6.2 mins, 45°, rotating
+ 6 kV / 425 μ A / 2.0 mins, 45°, rotating



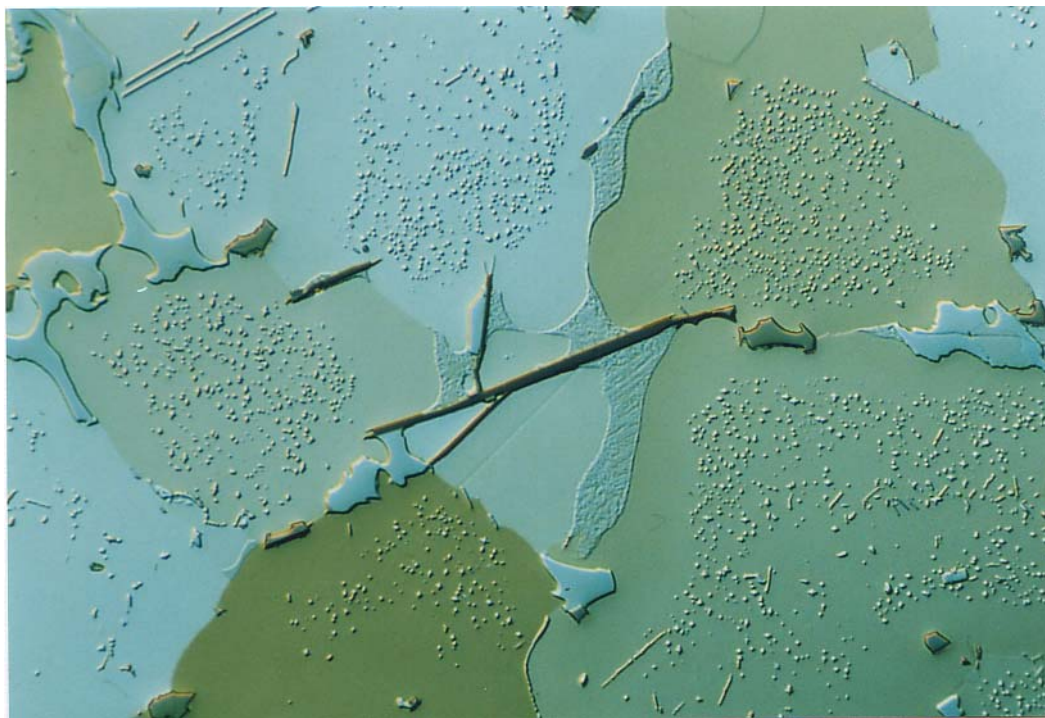
200x

Figure 77: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB125 (solder) after Bombardment with Ar Ions and additionally with Oxygen (Specimen 13), same position as Figure 76, Enhanced Contrast as a result of the Tarnish Layer, Parameter: Ar Etching (please see above) + O₂ Etching (8.5 kV / 445 μA / 3.4 mins, vertical, rotating)



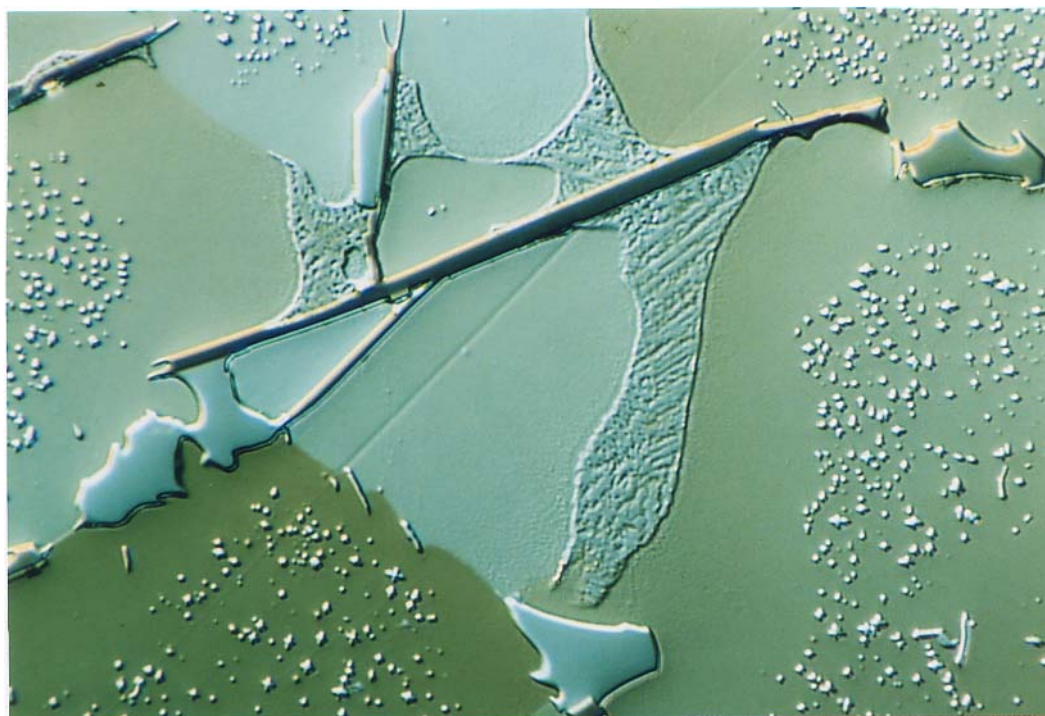
500x

Figure 78: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB125 (solder) after Bombardment with Ar Ions (Specimen 13), Micrograph with DIC, Slight Texturing of Base Material GW, Solder Matrix and Solder Phases, Section of Figure 76, Parameter: cf. Figure 76



500x

Figure 79: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB125 (solder) after Bombardment with Ar Ions and additionally with Oxygen (Specimen 13), Enhanced Contrast as a result of the Tarnish Layer, Enlarged Section of Figure 78, Parameter: Ar Etching (please see above) + O₂ Etching (8.5 kV / 445 μA / 3.4 mins, vertical, rotating)



1000x

Figure 80: Soldered Joint made of the Nickel Alloys Nimonic (base material) and NB125 (solder) after Bombardment with Ar Ions and additionally with Oxygen (Specimen 13), Enlarged Section of Figure 78, Enhanced Contrast as a result of the Tarnish Layer

3.1.9 Puddled Steel: Ferrite with Slag and Rust

Specimen 1

Etch gas Ar 6 / 435 / 8, 35°, rotating ⇒ Grain boundaries slightly etched, texture visible in the grain surface, iron oxide or rust slightly textured
(Specimen relatively high)

+ 6 / 435 / 7.2, 45°, rotating ⇒ Grain boundaries etching now o.k., textures in the grain surface decreased, iron oxide further textured, thereby at the same time lighter in color

Etch gas O₂ + 8.5 / 445 / 4.4, vertical, rotating ⇒ tarnish etching → **super**

Specimen 3 (Figures 81 to 84)

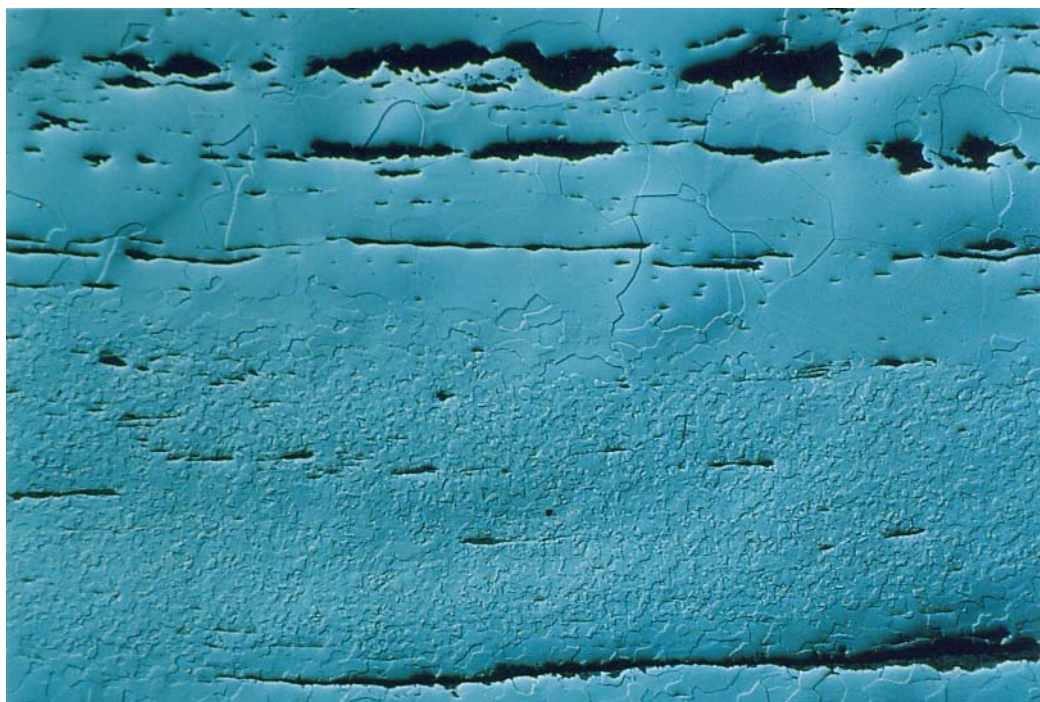
Etch gas Ar 6 / 435 / 8, 35°, rotating ⇒ Slight grain boundary etching (also in specimens which were not affected), iron oxide or rust textured
(Specimen relatively high)

+ 6 / 425 / 8, 30°, rotating ⇒ Grain boundaries etched further, in part martensite textures visible, rust etched for too long, now dark

Etch gas O₂ + 8.5 / 445 / 4, vertical, rotating ⇒ Oxides colored light blue (o.k.)
(Specimen off center)

Note:

The ion beam etching with argon develops the grain boundaries of the predominantly ferrite puddled steel. Iron oxide (rust) and oxidic slag are similarly textured. However the oxides are relatively dark, so that it is difficult to image them with high contrast. As a result of a supplementary bombardment with oxygen as the reaction gas, a layer is created, which when observed with a light microscope, appears to be light blue and can be imaged with high-contrast. The grain surfaces are different shades of brown depending on the orientation.



100x

Figure 81: Puddled Steel with Ferrite Matrix and Oxidic Slag after Bombardment with Ar Ions (Specimen 3), Ferrite Grain Boundaries Slightly Etched, Micrograph with DIC, Parameter: 6 kV / 435 μ A / 8 mins, 35°, rotating + 6 kV / 425 μ A / 8 mins, 30°, rotating

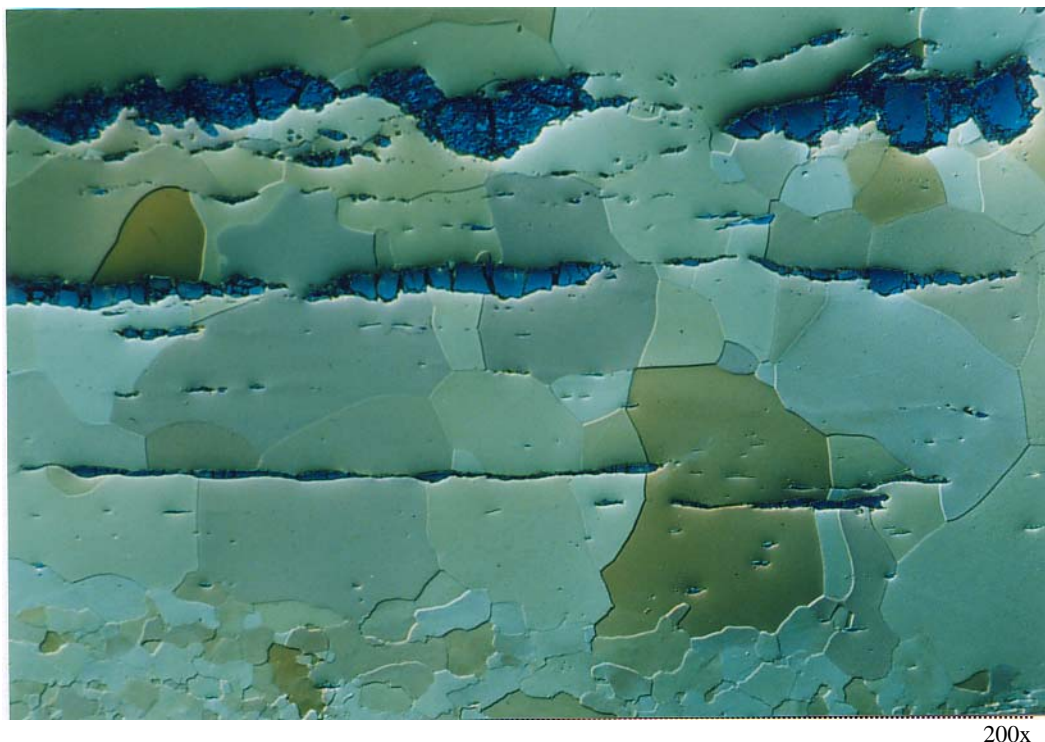


Figure 82: Puddled Steel with Ferrite Matrix and Oxidic Slag after Bombardment with Ar Ions and additionally with Oxygen (Specimen 3), same position as Figure 81, however Enlarged Micrograph with DIC, Enhanced Contrast as a result of the Tarnish Layer, Parameter: Ar Etching (please see above) + O₂ Etching (8.5 kV / 445 μA / 4 mins, vertical, rotating)

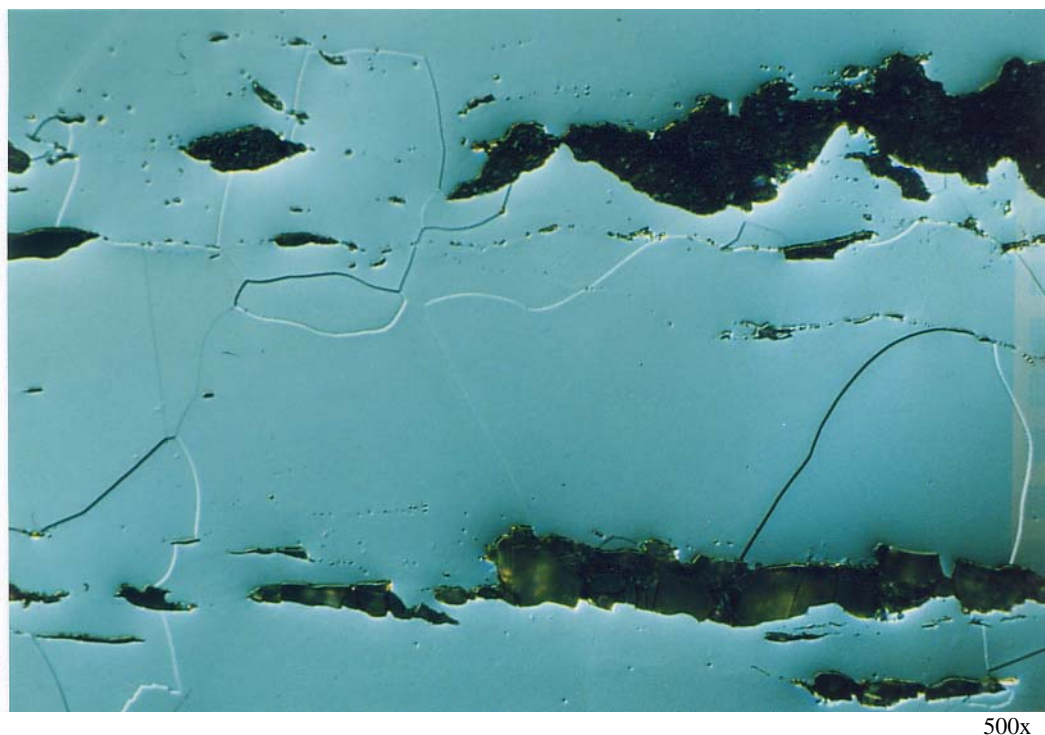
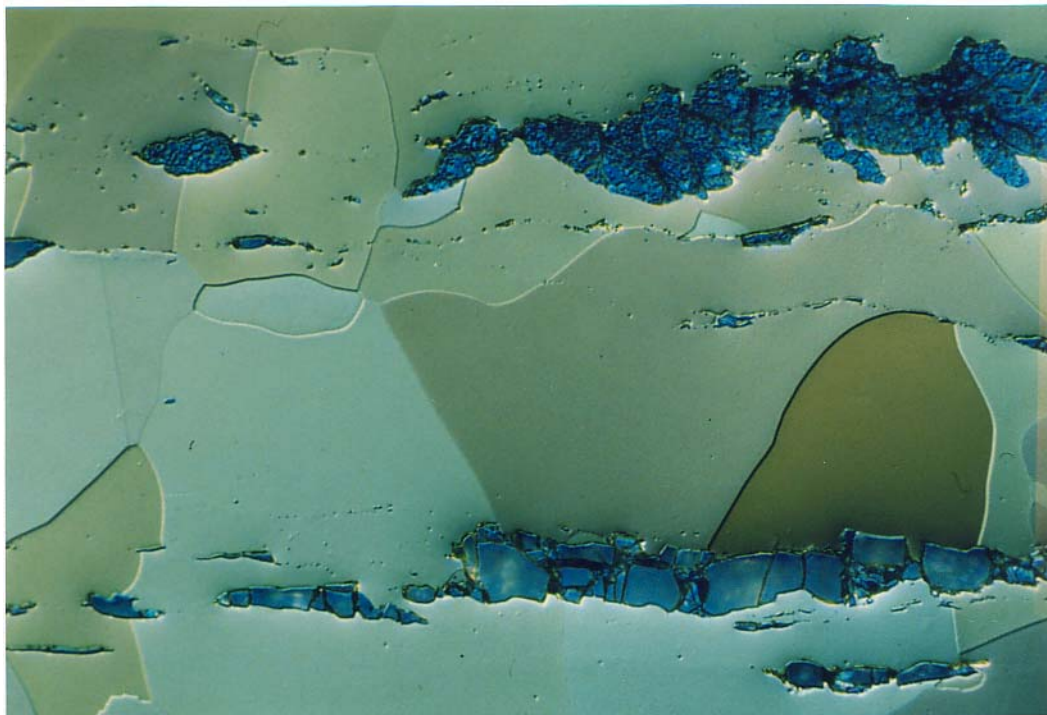


Figure 83: Puddled Steel with Ferrite Matrix and Oxidic Slag after Bombardment with Ar Ions (Specimen 3), Ferrite Grain Boundaries Developed, Micrograph with DIC, Parameter: 6 kV / 435 μA / 8 mins, 35°, rotating + 6 kV / 425 μA / 8 mins, 30°, rotating



500x

Figure 84: Puddled Steel with Ferrite Matrix and Oxidic Slag after Bombardment with Ar Ions and additionally with Oxygen (Specimen 3), same position as Figure 83, Micrograph with DIC, Ferrite Grain Boundaries, Enhanced Contrast as a result of the Tarnish Layer, Texturing of the Oxidic Slag clearly visible, Parameter: cf. Figures 81 and 82

3.1.10 Ceramics and Other Non-Metals

3.1.10.1 Aluminum Nitride AlN

Specimen 4	9.5 / 614 / 8, 37.5°, rotating	⇒ Grain boundaries slightly etched, in part the grain surface also etched, Y ₂ O ₃ similarly etched, not yet separated
	+ 9.5 / 614 / 2.4, 37.5°, rotating	⇒ Large protruding grains somewhat smoothed, apparently grain boundary etching, Y ₂ O ₃ similarly etched

Note:

The AlN specimens could only be assessed with difficulty as they were strongly relief-polished. From previous investigations, it is however a known fact that AlN can be textured without problems by ion bombardment. The etching angle should for this material not be too steep as then the Y₂O₃ is attacked more fiercely and can be separated off under certain circumstances.

3.1.10.2 PIC 151 [Pb(Zr_{0.52}Ti_{0.48})O₃] + 1-2% Ni, Sb (yellow)

Specimen L1	8.8 / 614 / 4, 30°, rotating	⇒ Grain boundaries already visible, however not all around the grains
	+ 8.8 / 614 / 4, 30°, rotating	⇒ Grain boundaries much better, specimen on one side tarnished in brownish color
	+ 8.8 / 614 / 1.2, 30°, rotating	⇒ Super etching
Specimen 15	8 / 435 / 8, 37.5°, rotating	⇒ Grain boundaries only half etched, i.e. not all around the grains visible
	+ 8 / 435 / 6, vertical, rotating	⇒ Grain boundaries now etched better, grain surfaces rough to grainy, quite good
	+ 8 / 435 / 3.2, 22.5°, rotating	⇒ Etching <u>very good</u>

3.1.10.3 PIC 141 [(Pb(Zr_{0.52}Ti_{0.48})O₃)] + ca. 1% Fe (black)

Specimen L4	8 / 450 / 2, 22.5, rotating	⇒ 5 mins interruption, specimen not taken out of the chamber
	+ 8 / 450 / 2, 22.5, rotating	⇒ both grain boundaries and 'cracks' visible
	+ 8 / 450 / 2, 30°, rotating	⇒ Grain boundaries and 'cracks' now somewhat more noticeable
	+ 8 / 450 / 4, vertical, rotating	⇒ Grain boundaries now better, mark in the center brownish in color
	+ 8 / 445 / 4, 22.5, rotating	⇒ Grain boundaries in the center of the specimen good, no cracks, → <u>quite good</u>
Specimen L6	8.8 / 614 / 4, 30, rotating	⇒ Grain boundaries already visible
	+ 8.8 / 614 / 4, 30°, rotating	⇒ Grain boundaries more visible
	+ 8.8 / 614 / 2, 30°, rotating	⇒ Grain boundaries even better, one-sided coating, Super etching!

Note:

This ceramic is very fine grained so that only after SEM (Scanning Electron Microscope) imaging a grain boundary etching could be established.

3.1.10.4 Zircon Oxide ZrO₂

Specimen 4	8 / 445 / 8, 45°, rotating	⇒ Grain boundaries slightly etched
	+ 8 / 445 / 8.3, 37.5, rotating	⇒ Grain boundaries further developed, pores slightly separated
	+ 8 / 445 / 3.4, 30°, rotating	⇒ Grain boundary etching quite good thus far

3.1.10.5 Al₂O₃ Standard

Specimen 1	9.5 / 600 / 4, 45°, rotating	⇒ Grain boundaries appear indicated (possibly relief as a result of polishing)
	+ 9.5 / 614 / 4, 45°, rotating	⇒ Certain grains are etched, also V-angle phase in the filling of the grains
	+ 9.5 / 614 / 4, 30°, rotating	⇒ in total the surface is etched, (evidence: material still there under particles of dirt)
	+ 9.5 / 614 / 6, 30°, rotating	⇒ Some grains further etched (for DIC acceptable),
	+ 9.5 / 614 / 4, 22.5°, rotating	⇒ Etching even better, however scratches enhanced after sputtering with AuPd → POL effect

Note:

The above-listed polished specimen is an exception among the oxide ceramics. Grain boundaries and, after sputtering with AuPd, a slight POL effect can also be observed. Possibly further parameter studies must be performed for ceramic materials, where appropriate using a reactive gas.

4. Concluding Remarks

After a brief, general introduction, those parameters were presented which had led to the best etching results being achieved with various materials. These data can quasi be used as introduction or guidance for new, unknown materials. Occasionally several etching methods were shown. In that way, it was intended to make it clear that the parameters can be varied without difficulty in order to achieve the aim. The etching process was interrupted sometimes on several occasion to monitor the progress of the structure development, for each material and each specimen responds somewhat differently and can quickly be over-etched. From that viewpoint, the etch tests should always be approached with caution.

5. Literature

Gräf, I.: *Ionenätzen - Stand und Perspektiven für die Gefügekontrastierung keramischer und metallischer Werkstoffe*; Praktische Metallographie, Band 35 (1998) Heft 5, 235/254 (Teil I), Heft 6, 316/326 (Teil II), Heft 7, 359/383 (Teil III)

Gräf, I.: *Anwendung des Ionenstrahlätzens bei Schichtverbundwerkstoffen*; Sonderbände der PM, Band 31 (2000) 349/354

Gräf, I.: *Ionenstrahlätzen von Verbundwerkstoffen mit der PECS*, Sonderbände der PM, Band 32 (2001) 351/354

Wechsung, R.: *Ionen-Ätzen - Der gegenwärtige Stand der Entwicklung*; Vakuuum-Technik, 26. Jahrgang (1977) Heft 8, 227/235

Acknowledgements

The etch tests were performed as part of the DGM-Arbeitskreis work on Ion Etching in Metallography (DGM Working Group). My thanks are due to the company GATAN GmbH, which made the PECS unit available for the comprehensive tests. Similarly I express many thanks to the company CARL ZEISS, from whom I was able to borrow the AXIOPHOT 2 for the light microscopic micrographs. I also wish to express my thanks to Professor Hartmut Fieß for his support with these tasks.