# Texture determination of thin Cu-wires by synchrotron radiation

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**Abstract.** A new method to investigate thin wires has been tested, which is based on a special sample holder and on a high energy X-rays. Due to the high penetration power of high energy X-rays quantitative texture data will be obtained without any additional corrections such as constant volume correction and absorption correction. The measurements have been carried out at the high energy beam line BW5 at HASYLAB – DESY (Hamburg). In order to overcome grain statistics problems on the investigated Cu-wire of 122µm thickness a special scanning routine together with the sample preparation allows to average over a wire length between 1mm and up to 240 mm.

## Introduction

Compared to sheets and extruded materials the investigation of wires is more complicated. This is due to the special sample shape particular in thin wires. Fig. 1a shows a typical Cu-wire of 1400 µm thickness. For conventional X-rays one has to use the reflection method after Schulz [1], because of the high absorption. The wire has to be cut in sections (Fig. 1b), which were fixed parallel on a sample holder (Fig. 1c). One can machine the sample to get a high quality surface that the standard Schultz method can be used or one had to correct for absorption [2, 3]. The preparation of a flat smooth surface by polishing was also used by Rajan and Petkie to investigate Cu wires by electron backscattered diffraction patterns (EBSD) [4]. Thermal neutrons and high energy X-rays both have a high penetration power for a large number of materials [5]. Depending on the beam flux, the grain size and on the wire diameter both radiations can analyze directly the wire as shown in figure 1a. To increase the grain statistics and to optimize the time consuming neutron experiment, one can put a set of wire sections in a vanadium cylinder (Fig. 1d) [6]. Sample preparation is very simple and corrections are not necessary, which has been used to investigate Fe-Cu composites [7].

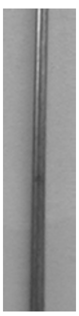


Fig. 1a: Cu-wire of 1400 µm Ø

Fig 1b: Cu-wire cut sections

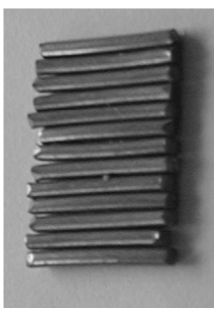


Fig 1c: Cu-wire set of parallel cut sections (X-ray)

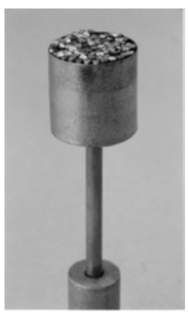


Fig 1d: Cu-wire Set of parallel cut sections (neutron)

Fig 1e: Cu wire of 122 µm Ø

Figure 1e shows an example of a thin Cu-wire of 122µm wire diameter, which gives some problems in the sample preparation. The neutron method is no more practicable for thin wires, because one has to collect to many sections in the vanadium cylinder to get reasonable counting times. Conventional X-rays show less correction for thin wires because of the wire diameter, but the sample preparation to get a parallel arrangement is complicated. Thus we decide to use a high energy X-ray beam and to develop a special sample holder fixing thin wires between 10 – 150 µm.

**Synchrotron texture measurement**

The synchrotron radiation is characterized by a wide range of available wavelength with a very high photon flux and an excellent brilliance. Due to the photon energy, hard X-rays with up to 200 keV show a high penetration power. In the case of copper the penetration depth to lose 50% of the incoming intensity is 0.015mm for Cu Kα- radiation, 2.0mm for 100 keV X-rays and 8.5mm for thermal neutrons. 2mm penetration depth is sufficient to measure a bundle of thin Cu wires or even a single wire. The X-ray technique at the high energy beam line BW5 follows the principle of a pinhole camera (Fig. 2). With its high brilliance one gets a highly parallel beam, which has to be directed to the sample. A typical beam size for texture measurements given by two automatic driven incoming slits is 1 x 1 mm² and a typical counting time for one exposure is in the order of some seconds. For complete pole figures one needs a set of individual exposures with different ω-angles. Δω depends on the sharpness of the texture. Blind areas as known from former film work [8], which have been used similar scanning routines, depend on the 2θ – angle of the measured pole figure. Due to the low wavelength of high energy X-rays the 2θ – angle is in the order of some degrees, so that blind areas don't occur. That means in the case of round samples one gets a complete pole figure without any corrections.

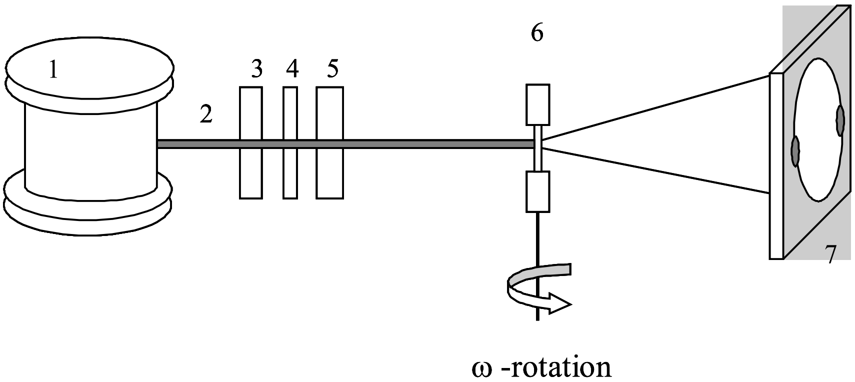
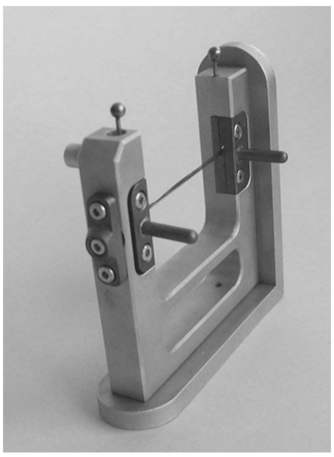



Fig. 2: Beam path at BW5 (1- monochromator hutch, 2- parallel X-ray beam, 3- incident slit, 4- diode, 5- incident slit, 6- sample, 7 – MAR345 image plate detector)



A special sample holder has been constructed (Fig. 3), which allows fixing up to ten wire sections parallel. Maximum length of a wire section is 5 mm. One can see in Fig.1e that thin wires tend to curl up so that a device for a soft stretching is necessary to get a sufficient composite sample. The sample holder can be used for wires between 10 – 150 µm. According to the small wire diameter and the curling effect the preparation needs a magnifying glass. Meanwhile a similar sample holder is available also for thin foils. Due to the geometry of the sample holder a restriction in ω-rotation is present, which did not influence the experiment because of the high texture symmetry.

Fig. 3: Wire sample holder with ten softly stretched Cu-wires

## Experiment

This method has been developed to characterize micro wires, which are essential components of microelectronic devices used in many industrial applications. The present example deals with a 99.9% copper wiredrawn to 122 µm wire thickness. A subsequent annealing leads to a coarse grained microstructure. Fig. 4a shows an image plate exposure taken in 2 sec., which was obtained with an entrance slit of 1x1 mm². The two inner rings represent Debye-Scherrer cones of the Cu (111) and the Cu (200) reflection. One can see clearly some single grains, which indicates an insufficient number of grains for a texture analysis using the volume method even for a bundle of 10 parallel wire sections. To overcome this problem an enlarged slit of 4x1 mm² along the wire axis was used. As results average information of about 40 mm wire length was obtained. In figure 4b one can see a much better grain statistics. Nevertheless the number of grains was still to low for a sufficient result.

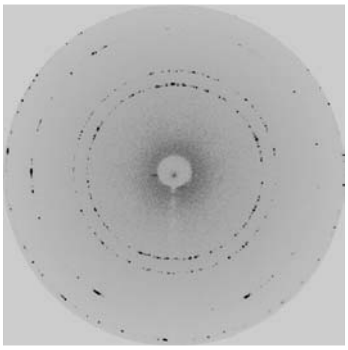


Fig 4a: Cu-wire measured with a 1x1 mm² slit (MAR345 image-plate exposure)

Fig 4b: Cu-wire measured with a 1x1 mm² slit (MAR345 image-plate exposure)

In order to increase the grain statistics a z-stage was used to measure the texture with Δ5 = 4 mm for 6 steps. Thus, a set of six pole figure data was produced to average over 240mm in wire length. One of the big advantages of the synchrotron beam was the high beam intensity so that relatively short counting times of two seconds were used. But on the other side this counting time was too short to run the integration along z automatically. The z-movement was too slow and consequently the set of six pole figure data was measured one after the other. Due to the readout time of Image-plate detectors the total counting time for one position was about 45 min. Table 1 summarizes the used measuring condition.

| | |
|---|---|
| Monochromator | imperfect Si |
| Beam-Energy | 100keV |
| Wavelength | 0.1024 Å |
| Sample to detector distance | 1130mm |
| Beam cross section | 4 x 1mm² |
| Exposure time | 2 sec. |
| z- movement | $\Delta z = 4$mm |
| ω-movement | $\Delta\omega = 2°$ |
| Cu pole figures | (111) and (200) |

Table 1: Measuring conditions for the investigation of a 122 µm Cu wire

For the data analysis pole figure information were filtered by a special program out of any image plate exposures. Due to the high penetration power one has only correct for a constant photon flux. Thereafter, these data have to be transferred to one of the standard input formats for a texture calculation. Figure 5 gives the Cu (111) and Cu (200) pole figures for all six sample positions. One can see a good agreement.

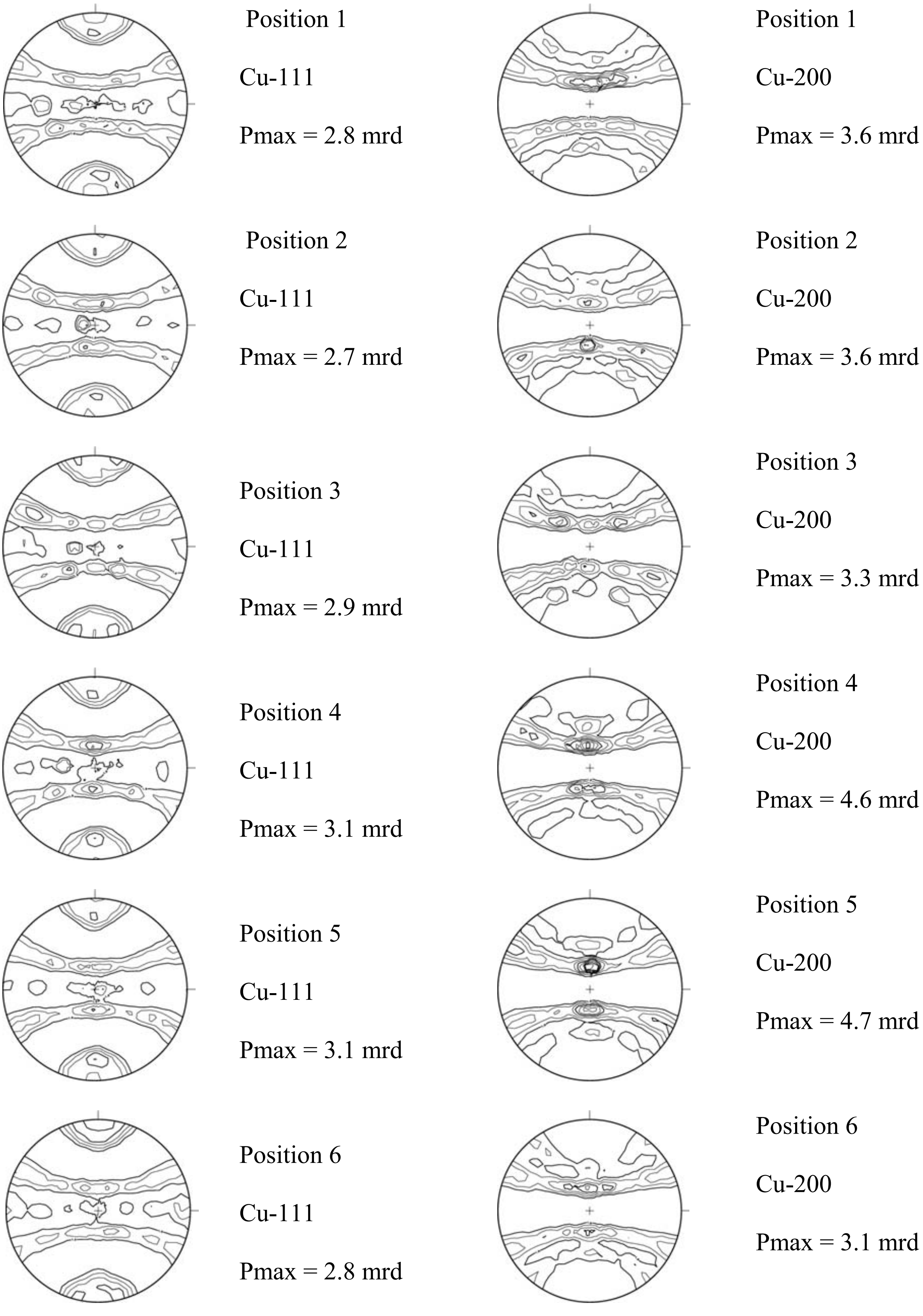
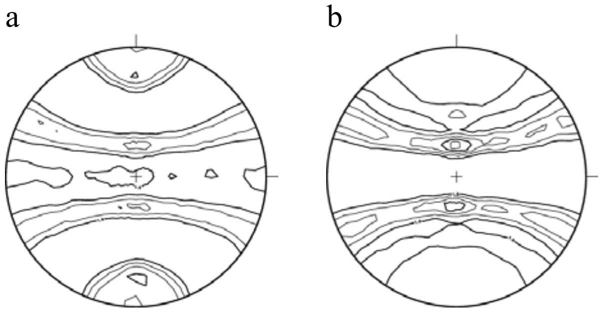



Fig. 5: Cu (111) and Cu (200) pole figures for all six positions

## Quantitative texture

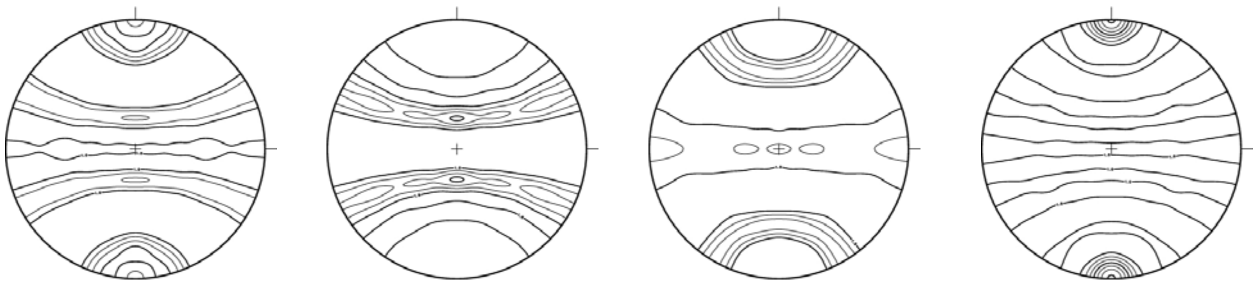
To get a good statistics the six Cu (111) pole figures and the six Cu (200) pole figures were added to a Cu (111) sum pole figure and a Cu (200) sum pole figure (Fig. 6a and 6b). As expected averaging results in a decrease of the pole density, this is more distinct in Cu (200) than in Cu (111). The quantitative texture (ODF) was calculated with the iterative series expansion method up to a degree of Lmax = 21. Figures 6 c – 6f show the recalculated Cu (111), Cu (200), Cu (200) and Cu (112) pole figures. Firstly a good agreement between measured and recalculated pole figures can be noticed. Secondly a fiber texture already indicated in the measured pole figures (see Fig. 5) is confirmed by the calculation. Thirdly the fiber axis in wire direction is close to the <112> direction (Fig. 6f).

a b



111 - Pmax 2.7 mrd 200 - Pmax = 3.2 mrd

c d e f



111 - Pmax 2.8 mrd 200 - Pmax = 2.6 mrd 220 - Pmax = 2.3 mrd 112 - Pmax = 4.1 mrd

Fig. 6: Measured (a-b) and recalculated Cu pole figures (c-f) averaged over 240 mm wire length

Cu-rods and Cu-wires show normally the typical double fiber with a stronger <111> texture component and a weaker <100> texture component. Contrary to the standard extrusion texture of fcc metals, the highly deformed and annealed 99.9% Cu wire of our experiment shows a fiber which is close to <112> (see Fig. 7), which has already be described by Wassermann and Grewen for the tension texture of copper [9]. A more detailed description of the quantitative texture will be given at another paper dealing with a set of Cu wires of different purity, of different annealing and of different wire diameter.

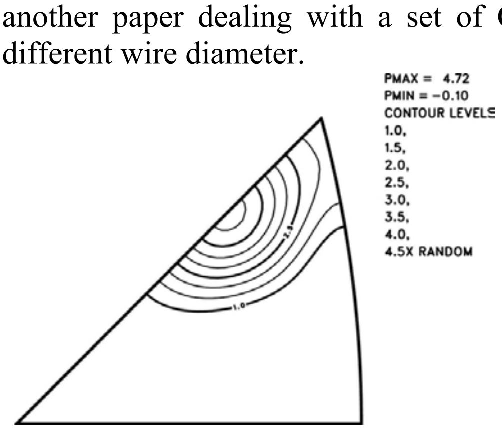



Fig. 7: Inverse pole figure in wire direction

**Summary**

Hard X-rays with a high penetration power are an excellent tool to characterize wires in transmission technique. In the case of copper wires of about 150µm or less the investigation on one hand of individual grains and on the other hand over 240 mm wire length was possible. A special sample holder allows bundling up to 10 wire sections of about 5mm in length so that the study can be carried without any additional sample treatment like etching or polishing. Moreover, correction for constant volume and absorption is not necessary. There are no limitations for other materials.

The investigated 99.9% pure Cu was part of a set of Cu samples with different purity, different wire diameter and different annealing. In the present example a fiber texture with a fiber axis close to <112> was observed. The typical double fiber of fcc metals <111><100> was not obtained.

**Acknowledgement**

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