SLAC-TN-06-008
August 2006

Search for Mechanically-Induced Grain Morphology Changes in Oxygen Free Electrolytic (OFE) Copper

Jennifer Sanders

Office of Science, SULI Program

Westminster College

Stanford Linear Accelerator Center

Menlo Park, California

August 18, 2006

Prepared in partial fulfillment of the requirements of the Office of Sciences, U.S. Department of Energy Science Undergraduate Laboratory Internship (SULI) Program under the direction of Dr. Robert Kirby of the Surface and Materials Science (SMS) department at Stanford Linear Accelerator Center.

Participant: ______________________________________
Signature

Research Advisor: ______________________________________
Signature

Work supported by Department of Energy contract DE-AC02-76SF00515

Table of Contents

ABSTRACT

Search for Mechanically-Induced Grain Morphology Changes in Oxygen Free Electrolytic (OFE) Copper. JENNIFER E. SANDERS (Westminster College, Fulton, MO 65251) ROBERT KIRBY (Stanford Linear Accelerator Center, Menlo Park, CA, 94025).

The deformation of the microscopic, pure metal grains (0.1 to > 1 millimeter) in the copper cells of accelerator structures decreases the power handling capabilities of the structures. The extent of deformation caused by mechanical fabrication damage is the focus of this study. Scanning electron microscope (SEM) imaging of a bonded test stack of six accelerating cells at magnifications of 30, 100, 1000 were taken before simulated mechanical damage was done. After a 2º – 3º twist was manually applied to the test stack, the cells were cut apart and SEM imaged separately at the same set magnifications (30, 100, and 1000), to examine any effects of the mechanical stress. Images of the cells after the twist were compared to the images of the stack end (cell 60) before the twist. Despite immense radial damage to the end cell from the process of twisting, SEM imaging showed no change in grain morphology from images taken before the damage: copper grains retained shape and the voids at the grain boundaries stay put. Likewise, the inner cells of the test stack showed similar grain consistency to that of the end cell before the twist was applied. Hence, there is no mechanical deformation observed on grains in the aperture disk, either for radial stress or for rotational stress. Furthermore, the high malleability of copper apparently absorbed stress and strain very well without deforming the grain structure in the surface.

INTRODUCTION

The Stanford Linear Accelerator Center (SLAC) remains on the forefront of particle physics with tools such as the two-mile linear accelerator. The SLAC linear accelerator has enabled scientists to make profound, Nobel Prize-worthy discoveries in the field of particle physics. Higher frequency, higher energy accelerators such as the International Linear Collider (ILC) brighten the horizon of future studies in this area.

Accelerator designs for producing higher energy beams require detailed and precise measurements during design and operation. The developmental Next Linear Collider Test Accelerator (NLCTA) at SLAC found that accelerator structures meeting the ILC design requirements experience excessive radio frequency (RF) breakdown rates at the desired 65MV/m accelerating gradient. With a scanning electron microscope (SEM), the post-operation locations of RF breakdowns were determined by the presence of visible craters in the copper structure [1]. Studies on the effects of these breakdowns show that RF breakdown has become one of several critical factors defining the limit of the operating gradient of accelerating structures [2]. Therefore, development of structure designs, fabrication procedures, and processing techniques that minimize structure damage are vital to the advancement of accelerator science.

Background

Linear Accelerators

The NLCTA consisted of many copper vacuum aperture disks, grouped as chambers that were manufactured from 99.9% pure Oxygen Free Electrolytic (OFE) copper. As electrons are accelerated in this copper structure, the chambers are exposed to an environment of radiation, electron and photon bombardment, and megawatts of RF power. Microwaves surge through the accelerator structure, creating oscillating electric and magnetic fields, forming an electromagnetic wave that travels down the inside surface of the accelerator. The electrons pass through each chamber at the proper wavelength to receive the maximum push from the electric field in that cell. Though copper is a relatively low melting point metal, its good electrical conductivity keeps the power losses of the copper low, making it a choice metal for the traveling wave structure. In order for the particle to receive the optimum boost, the inner surface of the wave structure must resonate at the proper wavelength. A smooth, contaminate-free inner surface ensures the desired acceleration.

Grain Deformation

The OFE copper is microscopically composed of pure metal grains from 0.1 to >1 mm in diameter. Understanding and determining the sources of grain deformation on the inside surface of the structures is essential to accelerator science. Previous studies show that RF breakdown is overwhelmingly responsible for grain deformation in the copper surface. Occasionally, the wave structure absorbed the power, accompanied by a surge of current, which left visible craters on the structure [2]. Multiple breakdowns in the same area of a malleable metal such as copper may have caused these small grains to deform, altering the consistency of the metal.

However, during the mechanical fabrication stage of autopsying NLCTA cells, the accelerator structure experienced a small rotational twist. The cutting tool, that separated cells, caught in the metal, jumped, and twisted the entire bonded stack of cells. The extent of grain deformation, if any, caused by this rotational damage to the structure is undetermined.

This paper addresses mechanical fabrication as a possible source of grain deformation, which may have contributed to the limited capability of the test accelerator. The mechanical fabrication damage simulated by manually twisting a bonded stack of accelerating cells has been examined and imaged by SEM for grain deformation. We are particularly interested in the grain structure of the cell planar iris, within several millimeters of the central aperture, where the electric field is highest and most breakdowns occur and deformed grains are formed. The irises are 2 mm thick and the overall thickness is 10 mm (Figure 1).

MATERIALS AND METHODS

Six OFE copper cells numbered 60 through 65 were bonded as a small test stack for this study (Figure 1). These cells are of the type used for fabricating the accelerator structure of the NLCTA, but for various reasons were not used for that purpose. In an accelerator structure, cell numbers reference cell location within the larger accelerator structure. This study uses the numbers solely for identification and comparison purposes. The individual cells were from a lot of cells rejected for previous use because of incidental handling artifacts, e.g. scratches and dents.

Each individual cell went through the annealing furnace and chemical cleaning before being diffusion bonded at 1015 – 1020^{o}C for an hour. Chemical etching on individual cells, lasting 60 seconds, effectively removed unwanted debris from the surface of the sample before bonding while the hydrogen furnace removes oxide from the surface area between the cells (which encourages atomic inter-diffusion between mating cell surfaces during the very high temperature bonding). After bonding, the stack of accelerating cells was vacuum-baked (to remove the excess hydrogen from the copper bulk) and then sealed in N_2 to maintain the clean surface. The bonded stack of accelerating cells measures approximately 52mm in length with cell 60 on the bottom of the stack, the recessed cup down (Figure 2).

For surface characterization before the mechanical damage, images of cells 65 and 60 (top and bottom) of the stack were taken with the SEM in magnifications of 30, 100, and 1000.

At these magnifications and a working distance of 15 mm (millimeters), the radial distance from the iris of the cell is easily observed and measured on the orders of 10, 100, and 1000 µm. Characteristic surface images of at least three different areas on the cell were filed for use in later comparisons.

To simulate the rotational damage done to the NLCTA accelerator structure during mechanical fabrication, the test stack was placed in a lathe (Figure 3), anchored on one end, and manually twisted 1.5º from the free end, severely damaging both end cells held by the lathe. The degree of twist was measured by the displacement of the number on the bottom cell from the centered number of the anchored cell. The rotated cell was displaced 1.25 mm—the desired amount of twist (2º – 3º).

To analyze the effect of the twist on each individual cell, the stack was cut apart. Fine machining smoothly shaved away as little as 25 µm per rotation until the wall around the recessed area was thin enough to tear away the iris. The iris of each cell was placed in a separate container labeled with the cell number. A small dot was also placed on the flat side of the iris of each cell to identify the accelerator beam out side, distinguishing one side from the other.

Imaging of cells 60 through 64 was done in the SEM with the set magnifications of 30, 100, and 1000. Characteristic images of multiple locations on the surface of the cells are analyzed for grain deformation at a resolution of 0.1, 1, and 10 µm and compared to the images taken of cell 60, recessed end, before the twist. The comparisons of the images taken before and after the twist provide the data for a correlation between mechanical fabrication damage and grain deformation.

RESULTS

SEM imaging was done on cell 60 (recessed end) of the bonded stack before the manual twist and cells 60 through 64 (recessed end) after the manual twisting of the test stack. Images of cell 60 before the twist generally appeared as in Figure 4: many grains varying in size, shape, and brightness. Figure 5 shows an image of the same location on the cell after the twisting. Though cell 60 underwent enormous exterior damage during twisting, comparisons with the images of this cell (Figure 4) taken before twisting at each magnification (30, 100, and 1000), show similarity in appearance and grain consistency.

Cells 61 – 64 were examined for changed morphology due to the manual twisting of the test stack. Figure 6 and Figure 7 show images of the grains in cells 61 – 64 on the scale of 1000 µm and 100 µm. The grain morphology is comparatively similar to the grains of cell 60 in which changes were not seen, even after extreme surface pressure. At the highest magnification used in examining the cells (1000X), multiple grains are observed to have high-angle grain boundaries, as in Figure 8. This type of boundary effects grains of different sizes, shapes and brightness throughout cells 61 through 64. Although the high-angle boundaries vary in severity, the deformation is only seen at the magnification of 1000 (10 µm scale) and therefore is not related to the macroscopic deformation searched for in this study.

DISCUSSION AND CONCLUSIONS

Because multiple occurrences of RF breakdown lead to grain deformation in the surface of OFE copper vacuum aperture disks, RF breakdown performance has become one of several critical factors limiting the capabilities of higher frequency accelerators. As a result great efforts have been made in characterizing, understanding, and reducing RF breakdowns [1]. However, searching for other possible sources of grain deformation is important for producing high energy, efficient accelerators.

This study focused on the SEM examination of five OFE copper cells after simulated mechanical fabrication damage, in order to correlate mechanical fabrication damage with grain morphology changes in the cells. SEM imaging of cell 60, before and after the compression of the lathe, showed that radial deformation of the cell does not induce changes in the morphology of the cell's grains within 2mm of the cell's iris (the area affecting the beam acceleration). Figure 4 and Figure 5 prominently show that grains remained the same, and furthermore, that the voids (dark spots) along the grain boundaries were not altered—evidence that the grains did not undergo any deformation.

Cells 61 – 64 lack evidence of rotational deformation from the twist performed on the stack. Figures 6 and 7 of the cells at magnifications of 30 and 100 resemble the previously discussed figures of cell 60 (Figures 4, 5).

The presence of high-angle grain boundaries (10 µm) results from the recrystallization process of copper after it was put through the furnace. When the copper cells are heated in the furnace, grains within the surface of the cell melt and merge together, forming bigger grains with smooth boundaries. During the cooling process, the grains recrystallize in this new formation nicely aligned within the copper structure. However, some grains do not merge with others or form compatible grain boundaries. This type of uncompromising behavior within the cell's surface results in high-angle grain boundaries. These high-angle grain boundaries are common in metallurgy and do not result from rotational deformation.

Therefore, because changes in grain morphology were not induced by either radial or rotational damage of a small degree, machine fabrication damage of this kind is uncorrelated with grain deformation within these cells. Furthermore, this study revealed the extensive ability of copper to absorb great stress and strain, as evidenced by the unchanged composition of the end cell (60) after the intense radial compression of the cell.

For future study of the effects of mechanical fabrication damage on the grain morphology of OFE copper, imaging of each individual cell prior to bonding the cells into a stack would help to diagnose grain deformation after twist or RF power. Also, the use of high-quality cells acceptable for accelerator use would confirm the results of this study.

ACKNOWLEDGEMENTS

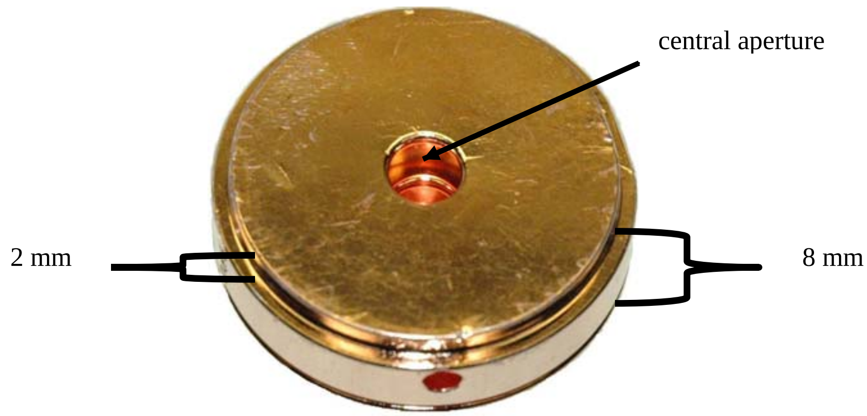
This research was conducted at the Stanford Linear Accelerator Center in Stanford, California. I thank the United States Department of Energy, Office of Science for giving me the opportunity to participate in the SULI program, acquiring amazing educational experience necessary for a future in science. The greatest appreciation goes to my mentor Robert Kirby for his excellent knowledge of the subject matter, and his desire to show me every aspect of lab work, as well as Dr. Frederic LePimpec for conceiving the project. I also thank Gerard Collet, who performed the manual twist and cutting of the cells, the other members of the SLAC Surface and Materials Science, and the Klystron Department (Chris Pearson and John Van Pelt) for providing the bonded structure.

REFERENCES

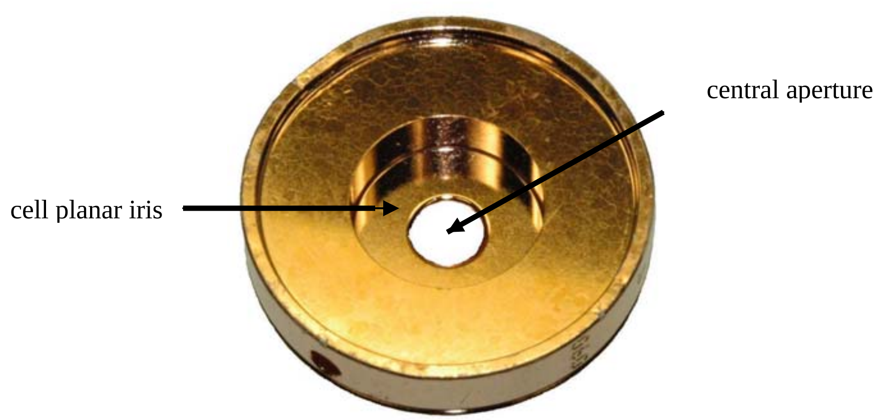
[1] Kristi Adamson, “The Effect of Mechanical Hardness on RF Breakdown,” August 2003, SLAC-TN-03006

[2] S.E. Harvey, F. Le Pimpec, R.E. Kirby, F. Marcelja, K. Adamson, E.L. Garwin, “Surface Analysis of OFE-Copper X-Band Accelerating Structures and Possible Correlation to RF Breakdown Events,” November 2003, SLAC-PUB-10197

FIGURES



(a) flat side



(b) recessed side

Figure 1 Flat side and recessed side of an OFE copper accelerator structure cell.

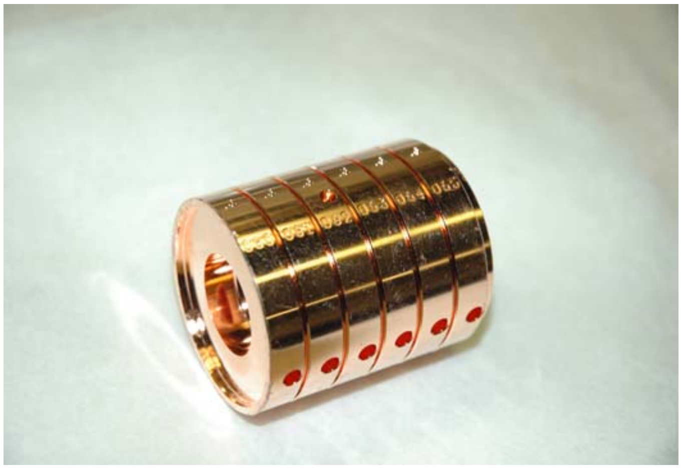


Figure 2 OFE Copper small test stack, cell numbers 60 through 65, used for this study before the manual twisting. The bottom of the stack, cell 60, shows the recessed cup of the cell. Notice the cell numbers and lower holes are perfectly aligned.

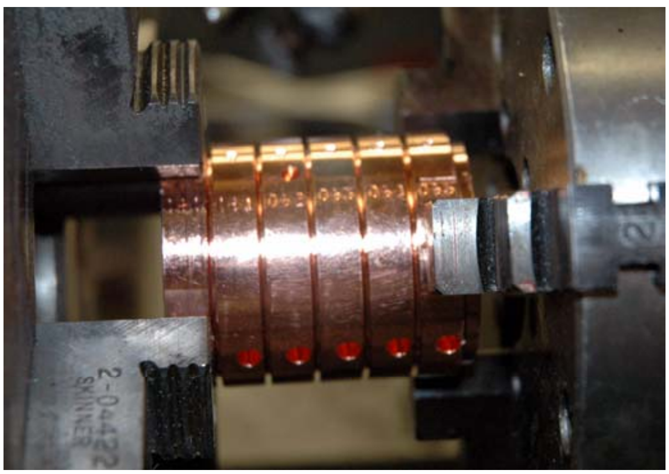


Figure 3 OFE Copper small test stack, cell numbers 60 through 65, locked in the lathe after the manual twisting. Notice the slight misalignment of the cell numbers as well as the lower holes.

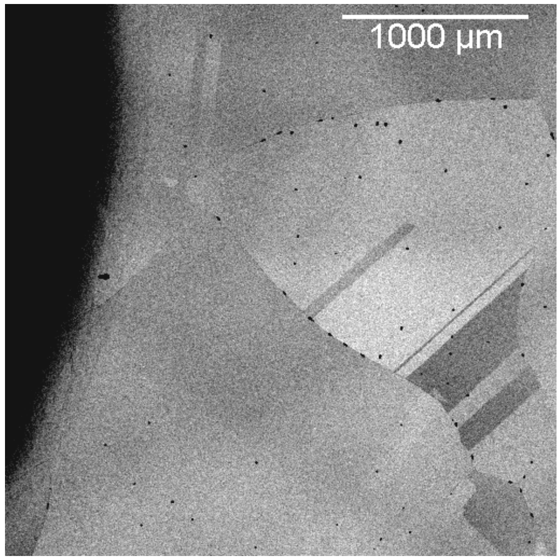


Figure 4 SEM image of copper grains in surface of cell 60 at magnification of 30X before twisting damage.

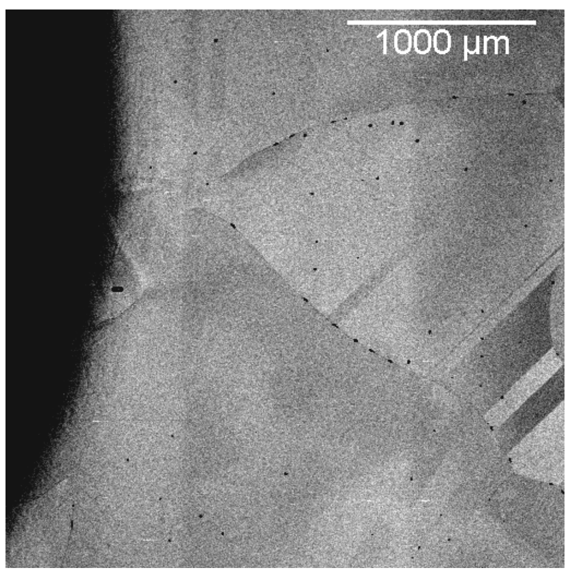


Figure 5 SEM image of copper grains in surface of cell 60 at magnification of 30X after twisting damage.

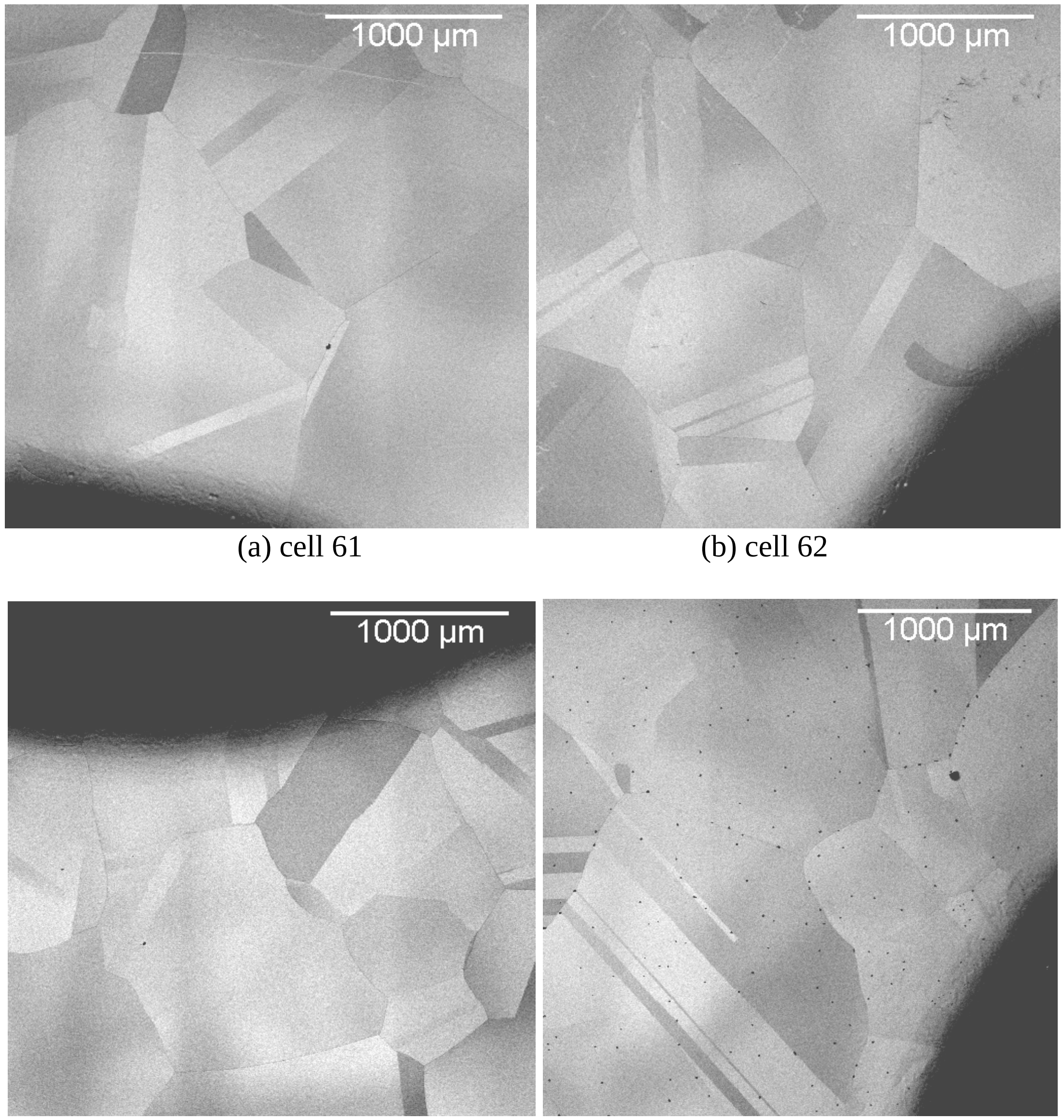
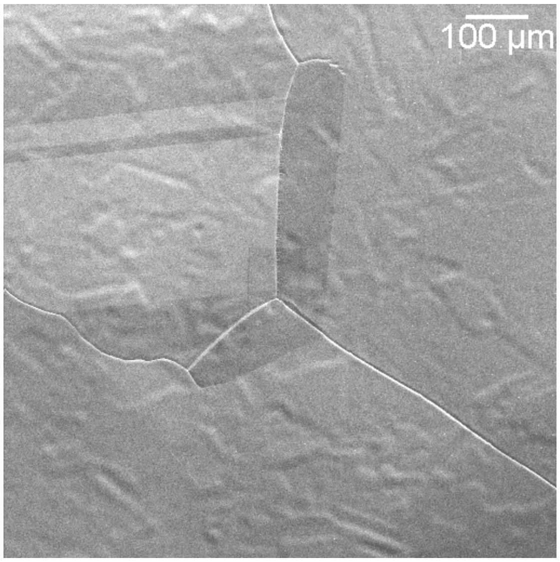
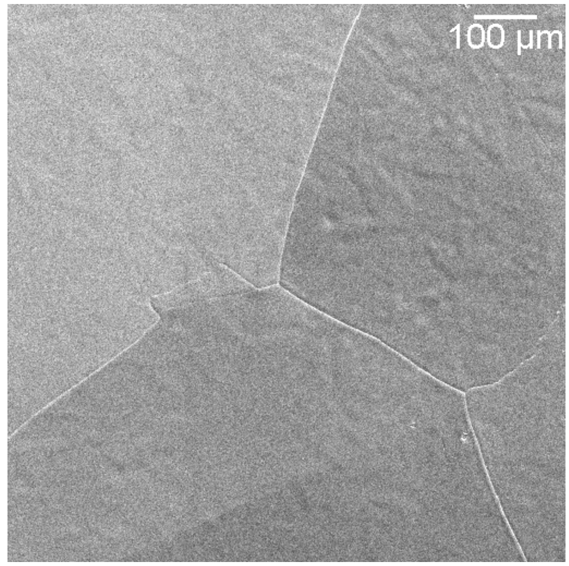


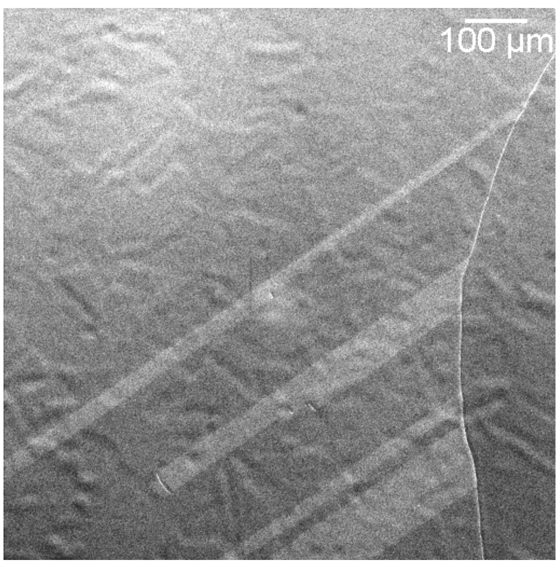
Figure 6 SEM images of copper grains in the surface of cells 61 – 64 at magnification of 30 after twisting. There are no signs of grain deformation.

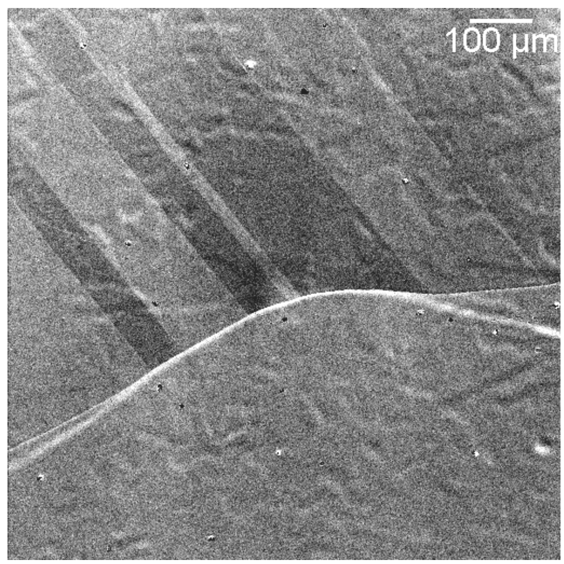




(a) cell 61

(b) cell 62





(c) cell 63

(d) cell 64

Figure 7 SEM images of copper grains in surface of cells 61 – 64 at magnification of 100 after twisting. There are no signs of grain deformation.

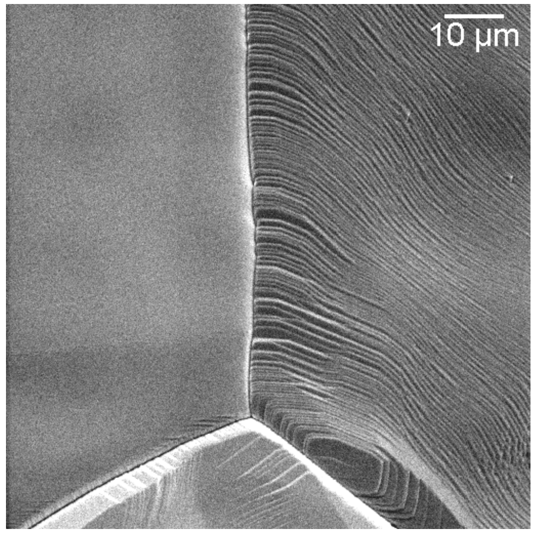


Figure 8 SEM image of high-angle grain boundary in cell 61at magnification of 1000 after twisting.