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Title:

Unusual Metal Behavior in Taylor Microwires

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Unusual Metal Behavior in Taylor Microwires

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Abstract

The objective of the research was to develop the Taylor microwire approach and employ it for fundamental studies of both ultra-fine-scale and non-equilibrium structures in selected metal and other materials. The Taylor microwire approach is an elegantly simple, experimentally convenient technique that combines both rapid solidification and deformation. In this technique, material is melted inside a glass tube and the softened glass with the molten material core is then drawn out into a fine microwire. Cu-16 at.% Ag microwires 10 μm in diameter were produced. Due to the rapid solidification occurring during the process, the microstructures of these microwires were metastable single phase, with an extremely small 10 nm grain size. Pure silicon microwires 10 μm in diameter were synthesized by the Taylor microwire technique. This is the first time such silicon microwires have ever been produced.

Background and Research Objectives

With the miniaturization of many engineering products, there is an increasing need for fine scale products such as tapes and wires with unique combinations of properties and high strength levels. There are a number of techniques based on vapor deposition of multilayer structures, rapid solidification, and wire drawing which have been used previously to produce suitable structures. However, an elegantly simple technique which combines both rapid solidification and deformation is the Taylor microwire technique of melting materials in glass tubes and drawing the composite to very small diameter wires (1,2).

The objective of the research was to develop the Taylor microwire approach and employ it for fundamental studies of both ultra-fine-scale and non-equilibrium structures in selected materials.

Importance to LANL's Science and Technology Base and National R&D Needs

The production of microwires by the Taylor method can be considered from two viewpoints. From a basic scientific viewpoint, it provides both ultrafine scale materials and new non-equilibrium structures in a form accessible to basic experimental studies to determine their properties. There is also a long term technological potential for microwires. They represent a relatively cheap fabrication route for fine scale wire products. Since they have electrically insulating glass coatings as a consequence of their production method, the microwires can be used for a variety of miniature electrical components including resistors, fast action fuses, thermocouples, and miniature motors. They may also have applications in electronics, MEMS, and biological systems.

Scientific Approach and Accomplishments

A schematic of the Taylor microwire approach is shown in Figure 1. This approach is an elegantly simple, experimentally convenient technique that combines both rapid solidification and deformation. In this technique, material is melted inside a glass tube and the softened glass with the molten material core is then drawn out into a fine microwire. Key aspects are that the working temperature of the glass be higher than the melting point of the material, and that the material not be reactive with the glass.

The Cu-Ag alloy system was selected for investigation, due to its model nature was well as the potential to produce high electrical conductivity/high strength microwires. Microwires of copper-silver alloys were successfully synthesized by the Taylor microwire approach. The Cu-Ag equilibrium phase diagram is shown in Figure 2. A composition of Cu-16 at.% Ag was employed. The Cu-Ag Taylor microwires were produced using Corning 7740 (Pyrex) borosilicate glass tubes, which have a softening temperature of 820 ^{o}C and a working temperature of 1250 ^{o}C. Sealed and evacuated glass tubes containing the Cu-Ag alloy were induction heated to the working temperature of the Pyrex glass and then drawn vertically. Cooling rates were estimated to be in the range of 10^{2}-10^{5} ^{o}C/sec, with the cooling rate increasing with decreasing diameter of the pulled Taylor microwire.

The finest diameter Cu-Ag microwire produced is shown in Figure 3. This microwire was 10 μm in diameter. A transmission electron micrograph of this Cu-Ag microwire is shown in Figure 4. Due to the high cooling rate (10^5 °C/sec), rather than being a two-phase equilibrium structure as indicated in Figure 2, this material was observed to be a metastable single phase material. Furthermore, the single phase grain size was extremely small, of the order of 10 nm, as shown in Figure 4. These results are described in detail in Publication 1.

A limited amount of Molecular Dynamics (MD) computer simulation was performed on the Cu-Ag microwire system. A Message-Passing Molecular Dynamics code developed specifically for very large scale (10-100 million atom) simulations was employed. Simulation of the deformation of a nanosized silver rod in a copper matrix is shown in Figure 5. The simulation indicates the generation of dislocations at the silver-copper interface during the incipient stages of deformation. Figure 6 shows a comparison of the predictions of the computer simulation to experimental transmission electron microscopy observations for the deformation of a nanosized silver rod in a copper matrix. Excellent correlation between the computer simulation and experimental results occurred.

A commercially available tensile testing stage for use in the Atomic Force Microscope (AFM) was obtained in the later stage of the Project. A photo of the tensile stage is shown in Figure 7. Unfortunately, Project funding limitations did not allow us to conduct tensile tests on Cu-Ag Taylor microwires.

The synthesis of pure silicon Taylor microwire was investigated. Since the melting point of pure Si is 1410 °C, a higher temperature Taylor microwire glass heating system had to be developed. The glass employed was Corning 7913 high silica (Vycor), which has a softening temperature of 1530 °C and a working temperature of approximately 1900 °C. An optical floating zone single crystal growth system was employed to heat the Vycor glass to its working temperature, so that the Taylor microwire could be pulled. Pure Si chunks were loaded into evacuated and sealed Vycor glass tubes. These Vycor tubes were then heated to approximately 2000 °C using focused halogen lamp heating, and the silicon Taylor microwire synthesized.

Figure 8 shows the pulled morphology of the silicon microwire. Due to a fluid instability during the pulling of the molten glass, two parallel silicon microwires were synthesized. Figure 9 shows the maximum continuous length of silicon microwire that was produced on the Project. A continuous length of 46 cm was produced. The continuity of this length was verified by measuring the electrical resistance of the full 46 cm microwire length. Figure 9 also illustrates that, because of the very fine diameter of the microwire, it possesses considerable flexibility under elastic strain conditions. Figure 10 shows the cross-section of the silicon microwire, and indicates its very fine size. Figure 11 shows transmission electron micrographs of the silicon microwire. The microwire was observed to be polycrystalline, with dislocation substructures in evidence. As expected, there was no reaction layer at the silica-silicon interface. A Patent Application has been filed based on these results, and a paper is in the process of being submitted for publication.

Publications

1. Han, K., Embury, J.D., Petrovic, J.J., and Weatherly, G.C., "Microstructural Aspects of Cu-Ag Produced by the Taylor Wire Method", Acta Mater., **46**, 4691-4699 (1998).
2. Patent Application S-94,619, "Semiconductor Microwires", Inventor: John J. Petrovic, filed with the U.S. Patent and Trademark Office on 9 June 2000.
3. Petrovic, J.J., Hoover, R.C., Field, R.D., and Han, K., "Silicon Microwires", in preparation for the Journal of Materials Research.

References

1. Taylor, G.F., "A Method of Drawing Metallic Filaments and a Discussion of Their Properties and Uses", Phys. Rev., **23**, 655-660 (1924).
2. Donald, I.W., "Review: Production, Properties and Applications of Microwire and Related Products", J. Mat. Sci., **22**, 2661-2679 (1987).

Figure Captions

Figure 1: Schematic of the Taylor microwire process.
Figure 2: Copper-silver equilibrium phase diagram.
Figure 3: 10 μm diameter Cu-16 at.% Ag Taylor microwire.
Figure 4: Transmission electron micrograph of Cu-16 at.% Ag Taylor microwire. The microstructure is a metastable single phase, with a grain size of 10 nm.
Figure 5: Molecular Dynamics simulation of the deformation of a silver rod in a copper matrix. The generation of dislocations at the silver-copper interface is indicated.
Figure 6: Comparison of Molecular Dynamics computer simulation predictions to actual experimental transmission electron microscopy observations for the deformation of a nanosized silver rod in a copper matrix. Excellent correlation between simulation and experiment is observed.
Figure 7: Tensile testing stage obtained for the Atomic Force Microscope (AFM).
Figure 8: Morphology of pure silicon Taylor microwire pulled using a Vycor glass tube.
Figure 9: 46 cm continuous length of silicon microwire. Note the elastic flexibility.
Figure 10: Cross-section of silicon microwire.
Figure 11: Transmission electron micrographs of silicon Taylor microwire.

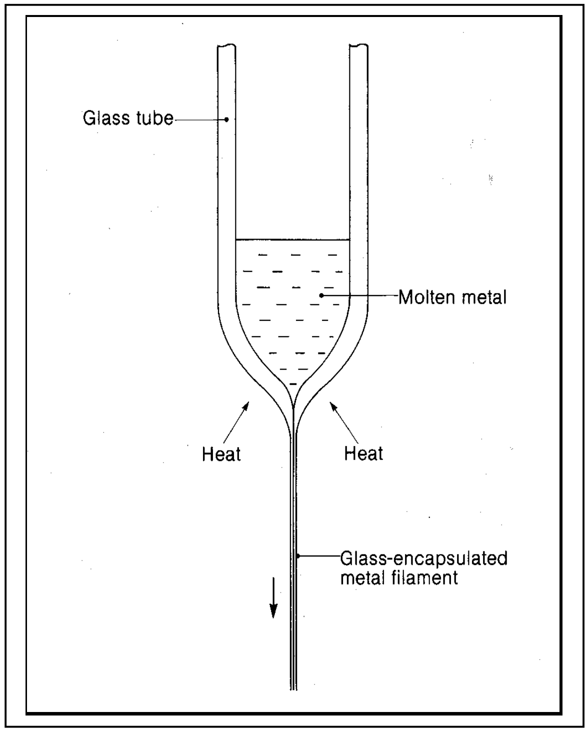

Figure 1: Schematic of the Taylor microwire process.

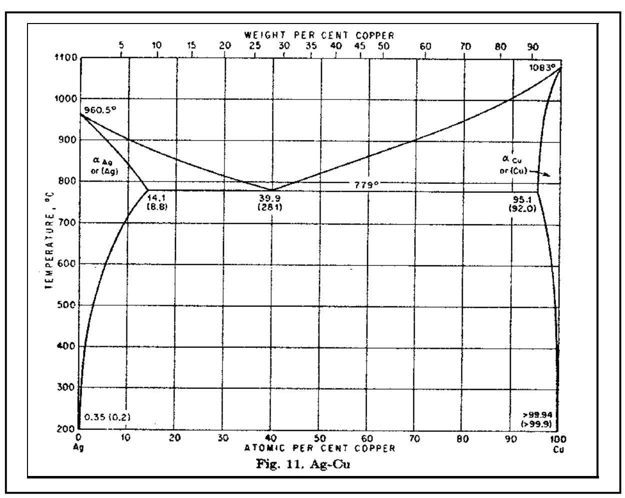

Figure 2: Copper-silver equilibrium phase diagram.

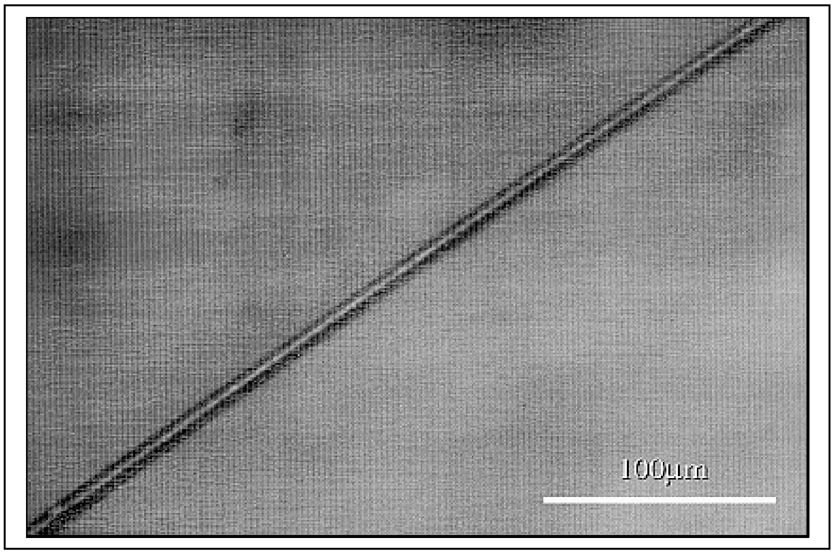

Figure 3: 10 μm diameter Cu-16 at.% Ag Taylor microwire.

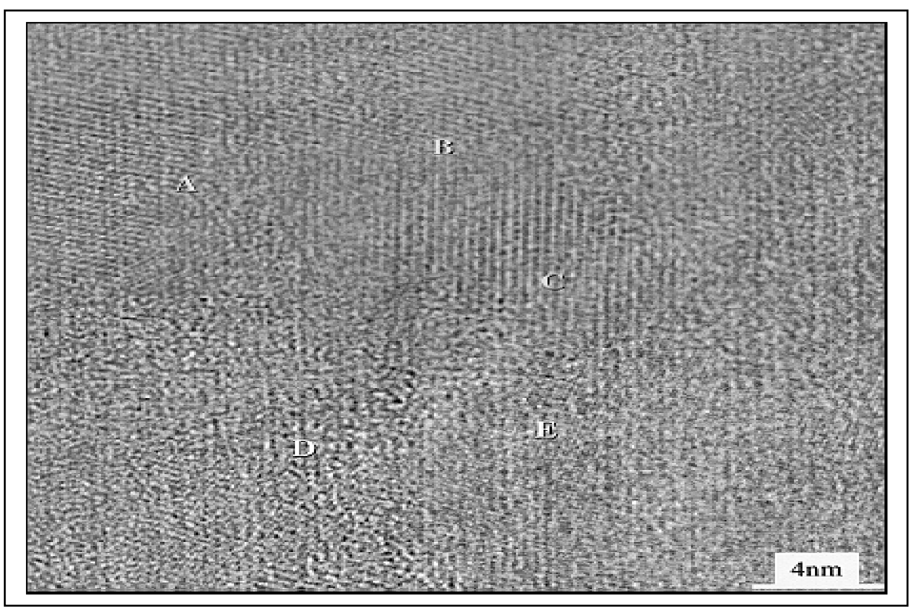

Figure 4: Transmission electron micrograph of Cu-16 at.% Ag Taylor microwire. The microstructure is a metastable single phase, with a grain size of 10 nm.

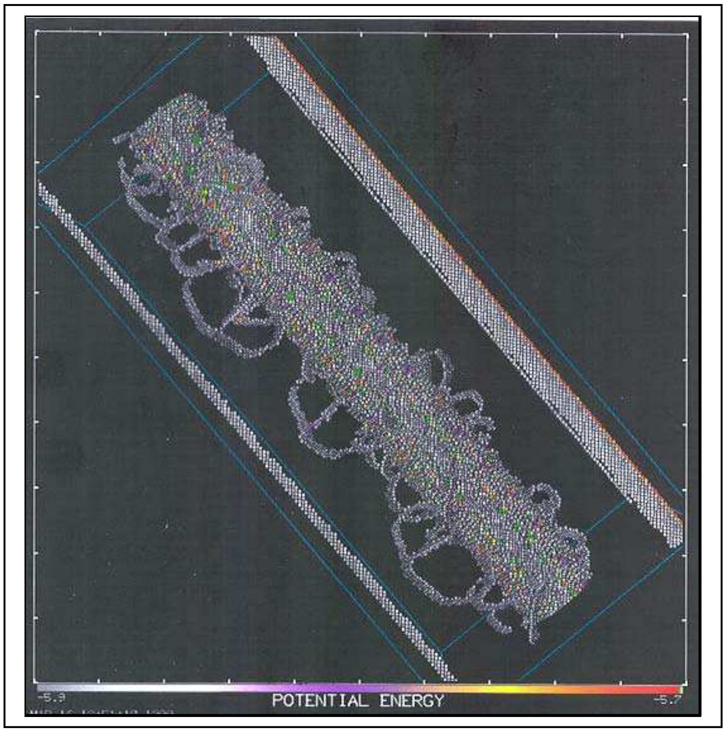

Figure 5: Molecular Dynamics simulation of the deformation of a silver rod in a copper matrix. The generation of dislocations at the silver-copper interface is indicated.

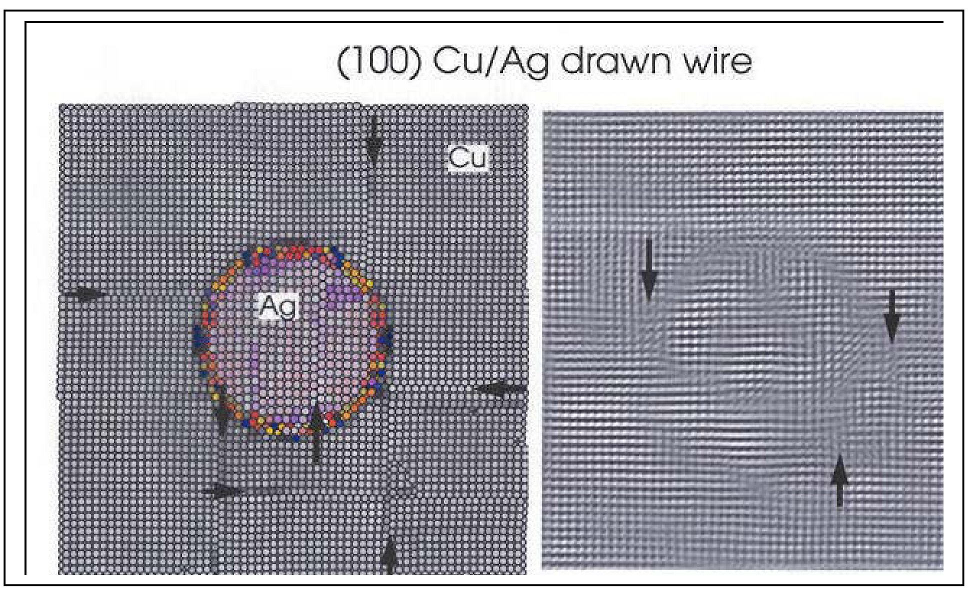

Figure 6: Comparison of Molecular Dynamics computer simulation predictions to actual experimental transmission electron microscopy observations for the deformation of a nanosized silver rod in a copper matrix. Excellent correlation between simulation and experiment is observed.

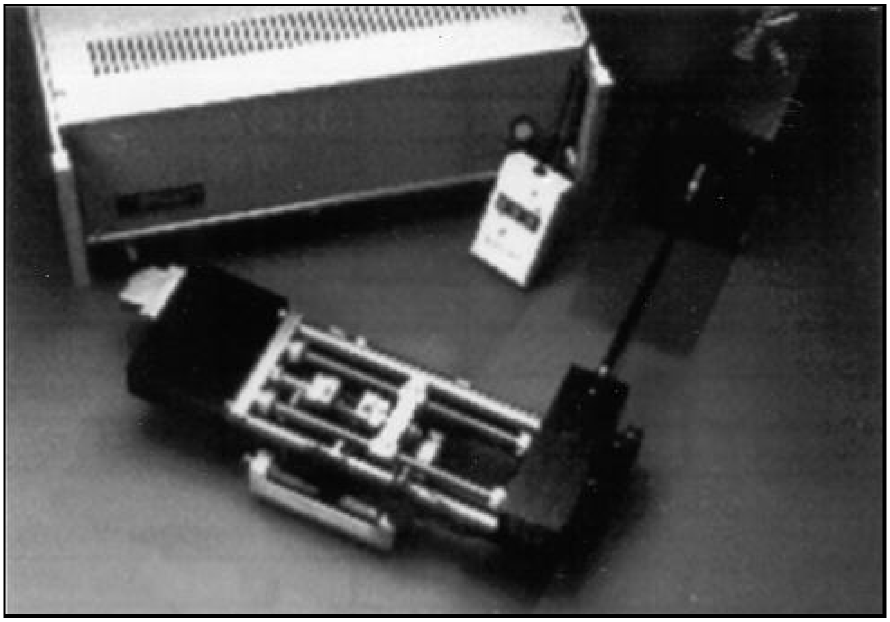

Figure 7: Tensile testing stage obtained for the Atomic Force Microscope (AFM).

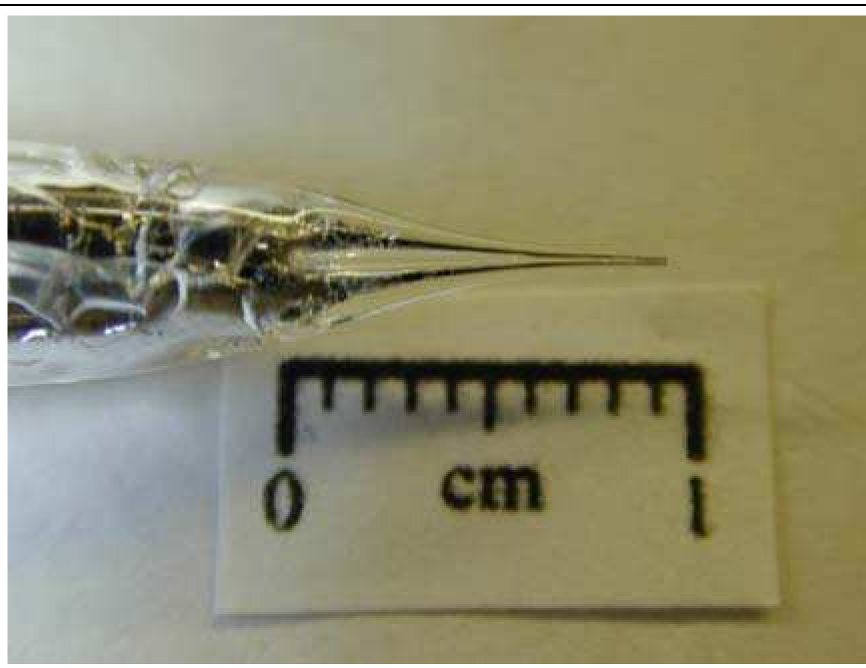

Figure 8: Morphology of pure silicon Taylor microwire pulled using a Vycor glass tube.

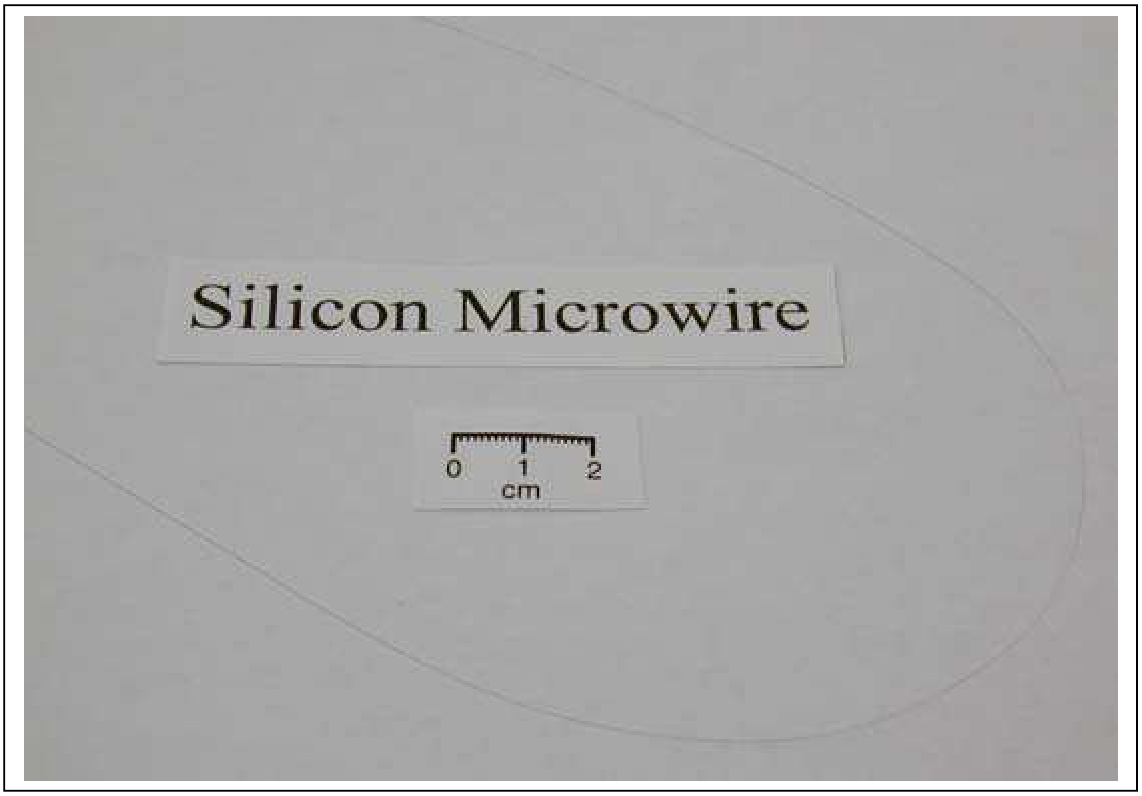

Figure 9: 46 cm continuous length of silicon microwire. Note the elastic flexibility.

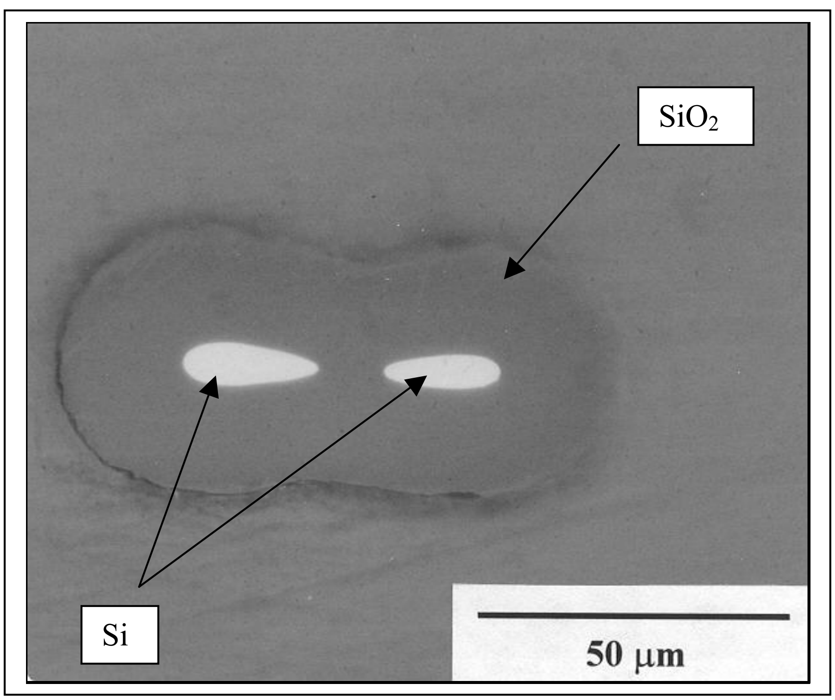

Figure 10: Cross-section of silicon microwire.

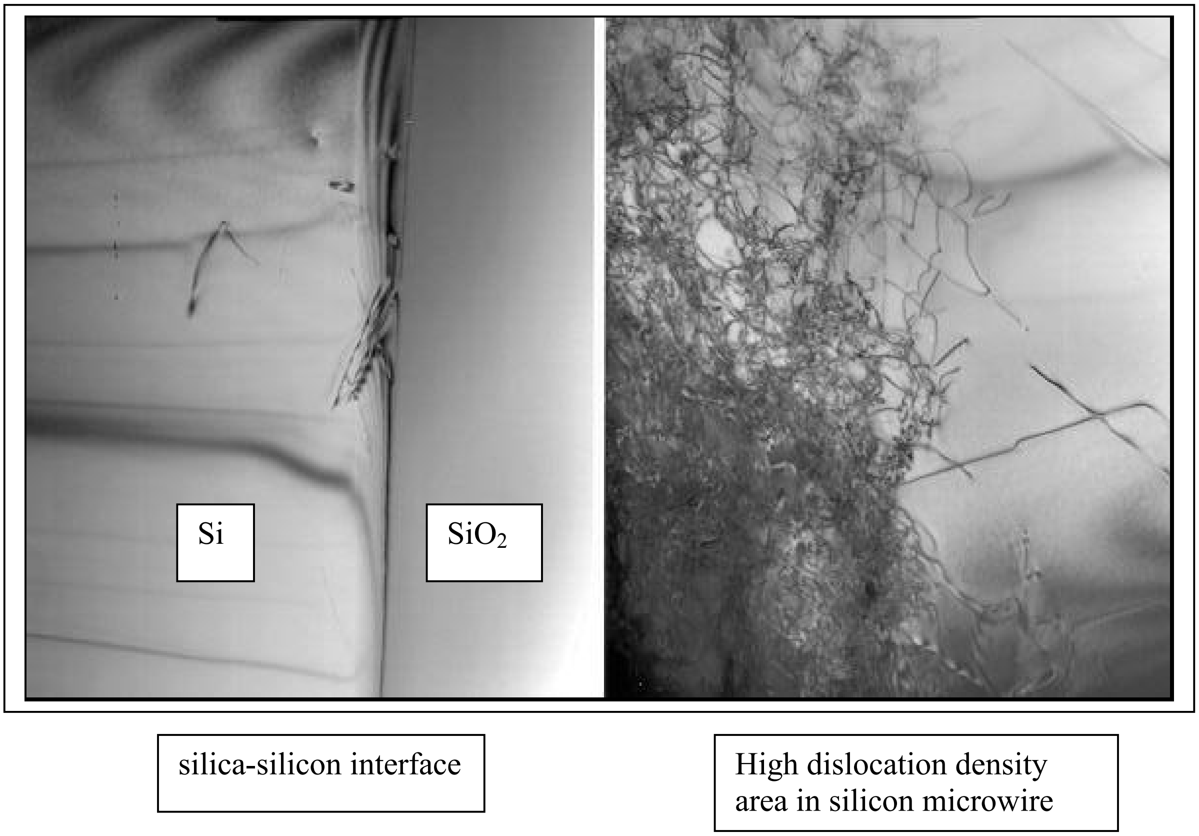

Figure 11: Transmission electron micrographs of silicon Taylor microwire.