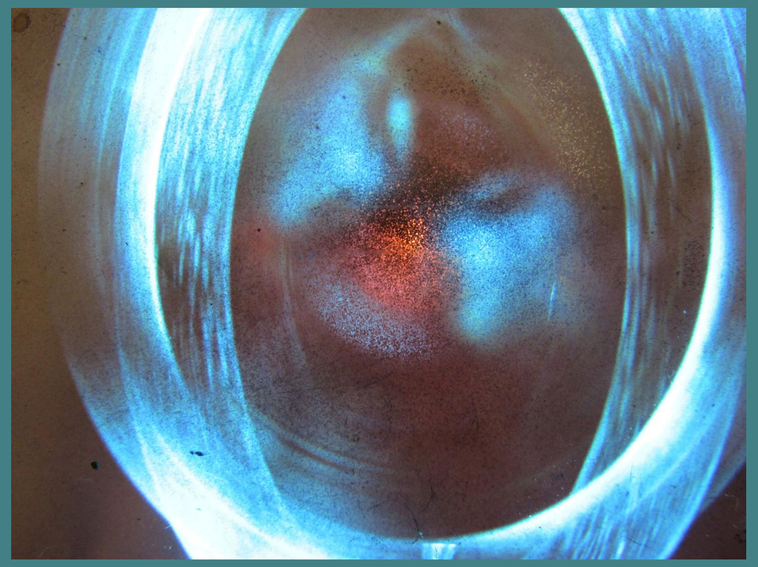

Field Electron Emission of Hafnium Carbide

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Abstract

Hafnium carbide, or HfC, is a robust material whose high melting point makes it an advantageous electron source for a variety of uses, including imaging. This research focused on the effects of an applied field at high temperature on HfC crystals, which induces a faceting effect over time. The cause of this faceting was studied to determine whether it could be correlated to evaporation or self-diffusion during use. The method for electrochemically etching hafnium carbide cathodes was also optimized, and the crystallographic geometry of the tip surfaces was studied using field emission microscopy.

Introduction

In many fields of scientific research, empirical evidence can be significantly strengthened by the utilization of electron microscopy. As advancements in electron microscopy bring sharper resolution and higher magnification, it is important that the emission sources producing these images be refined and their properties understood.

Hafnium carbide is a relatively new material to the microscopy industry. It is a transition metal carbide, which are known to perform well in relatively poor vacuum environments due to their extreme hardness and low work function. This opens opportunities to use these sources as field emitters in a variety of atmospheres where conventional cathodes would fail.

Single crystal HfC field emitters are utilized in this research, with intention of studying how the cathodes wear over time, as well as structural characteristics of the material.

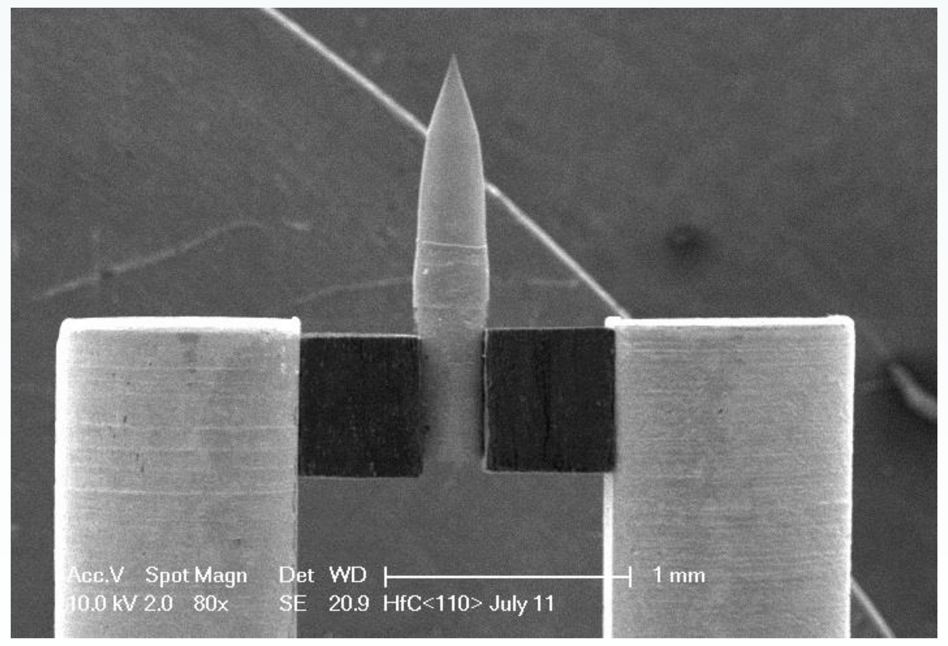


Fig. 1: an etched and mounted crystal

Electrochemical Etching

The process of etching a carbide crystal is necessary to achieve a tip with a small enough radius that will properly emit an electron beam. Hafnium carbide crystals have particularly small tip requirements, ranging from between 50 nm to 250 nm radius (see Fig. 2). Because of their disappearingly small tip and the extreme hardness of the material, etching this material is a meticulous process in which precision is necessary.

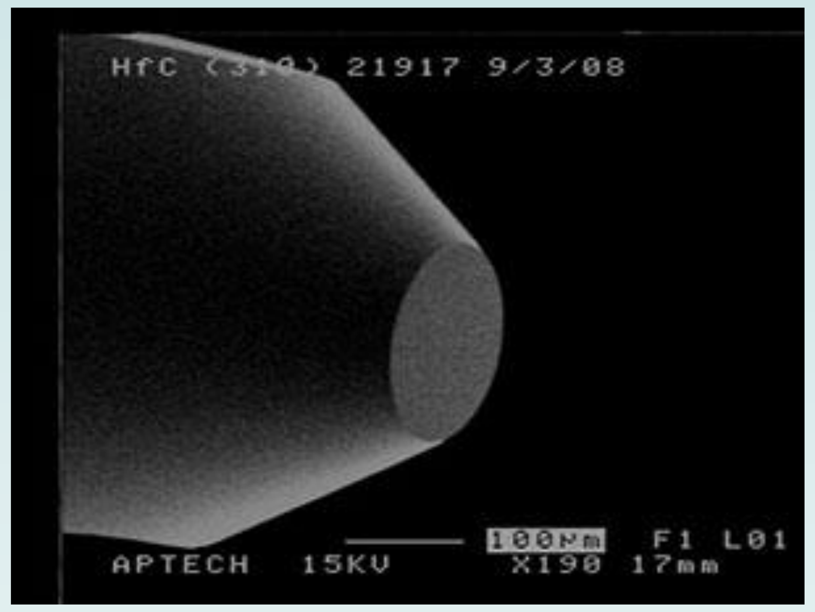


Fig. 2: SEM image of an etched HFC tip

The material used for etching begins with purified single crystal stock of the desired plane orientation. A chemical solution is composed of one part acetic acid to nineteen parts perchloric acid. The material is mounted and hooked up to a power supply. A mounted tantalum etching ring is then submerged in the etching solution, which then forms a drop of solution inside the ring. The crystal is then dipped into that drop, and voltage is applied. Repeated dipping of the ring and varying pulse length will etch away at the material, eventually causing an hourglass shape to form. This produces a teardrop shape to also form below the hourglass (see Fig. 3). While maintaining the size of the teardrop, the apex of the waist is slowly decreased until the teardrop falls off the end of the material, resulting in a disappearingly small tip (see Fig. 1). Premature drop off or prolonged pulse length will result in a jagged or oversized tip.

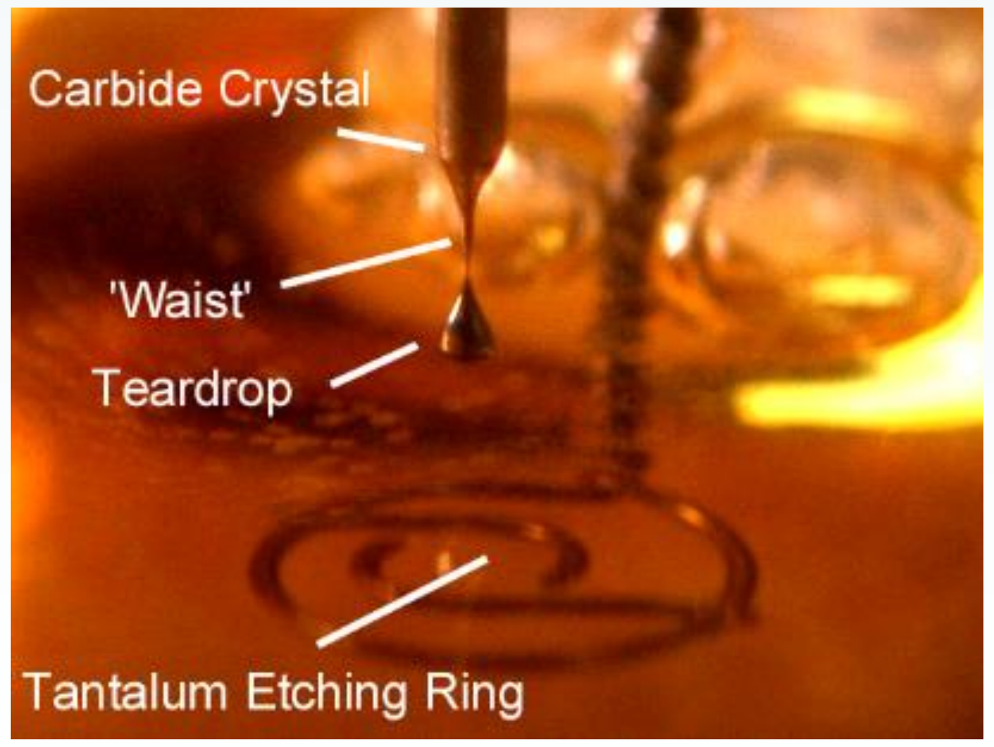
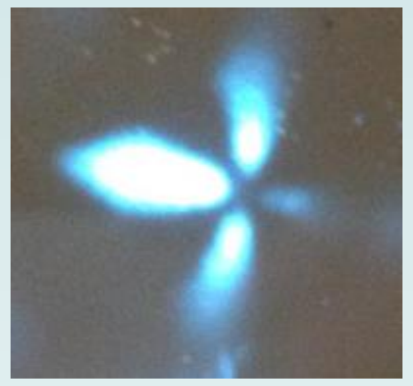


Fig 3: etching process

Crystal Plane Orientations

When used as an emission source, a hafnium carbide cathode emits a beam of electrons from the surface of its etched tip. The tips of these crystals can be characterized by different planes. These planes act like a three-dimensional lattice structure that, depending on how the crystal is oriented on that lattice, will change the emission properties of the crystal. For example, the two figures below display a <110> oriented crystal and a <210> oriented crystal. Work function of a crystal is plane-dependent, resulting in HfC<110> having a higher work function than HfC<210>. Consequently, HfC<210> will be a more optimal orientation to use as an emission source.

HfC<110>



HfC<210>

Field Electron Microscopy can be used to study the plane orientation of these materials, as seen above. Mounted in a vacuum system, these thermionic projections can be observed as a result of current and high voltage through the crystal, which projects the surface geometry onto a phosphorous screen at which the crystal is aimed. It is important to know the surface structure of emission crystals to ensure quality and performance under an applied field.

This procedure was done not only to study the characteristics of different crystal geometries, but to study how the applied field at high temperatures changes the structure of the tip over time.

Problem Faceting of HfC

At high temperatures, an applied field on HfC tips was found to induce a faceting affect on the surface geometry of HfC crystals (see Fig. 4). While it does appear to be a function of an applied field and high temperatures, the reason of this affect is not yet known. It is theorized to be caused by either evaporation or self-diffusion. This provokes more research questions, for example whether changes in the composition of the crystal itself could prevent the faceting from occurring.

Additionally, faceting has only been found to occur on the <100> and <111> planes. Because the <100> plane is known to have the lowest surface free energy, it is theorized that faceting is prone to occur here because it is easier for the material to cleave on those planes. This does not, however, explain why faceting also occurs on the <111> plane, which has a higher surface free energy.

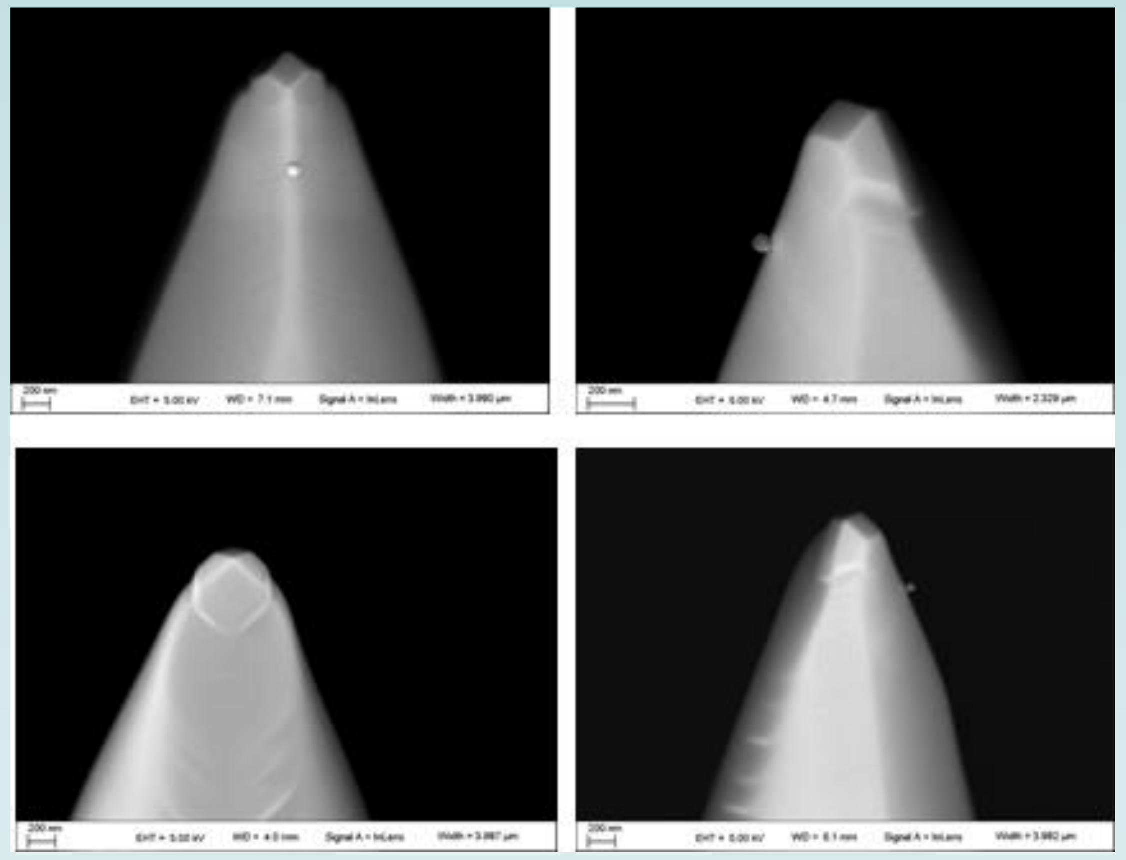


Fig. 4: examples of faceting on HfC crystals

Conclusions

Current results suggest that faceting of hafnium carbide is more prevalent on plane orientations with lower surface free energy. Refinements to the electrochemical etching process allow more consistent crystal characteristics and tip diameters to be used to compare in future studies of this. In these studies, the time duration of the applied field at high temperature is to be kept consistent, and the orientation of the crystal is to be taken into account.

The next step of this ongoing research project is to further induce faceting on specific plane orientations, and quantify the amount of deformation over time. With a refined focus on the <100> and <111> planes, more tests will be done to investigate the cause of this faceting and its possible relations to surface free energy. Factors of evaporation and self-diffusion will also be studied to determine how the facets are forming.

Acknowledgments

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