Using XRD and TGA-DSC to Characterize a Silicon Carbide Powder

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Introduction

Silicon Carbide (SiC) is a ceramic commonly used in abrasives, electrical components, and high temperature applications due to the material's high level of hardness, temperature resistance, and corrosive resistance[1] (Fig 1). These mechanical properties of SiC are what make it so versatile and commonly used. However, there is little understanding behind the underlying thermodynamic principles that dictate SiC's mechanical properties[2]. Further understanding these principles could allow for the creation of SiC with improved properties so that it can be optimized in the context of an intended application.

To further develop an understanding of SiC's thermodynamic properties, a high purity sample must first be attained in order to perform calorimetric experiments. A sample must first be characterized in order to ensure its purity. This project uses X-Ray Diffraction[3] (XRD) and thermogravimetric analysis differential scanning calorimetry[4] (TGA-DSC) to characterize an industrial sourced SiC sample in order to determine its composition and purity.

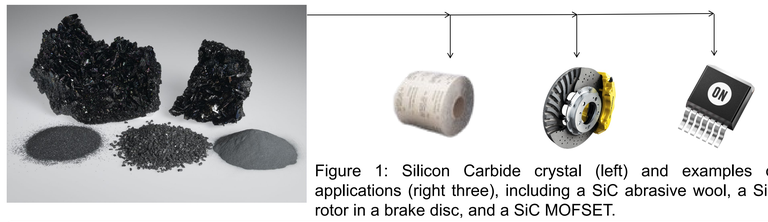

Figure 1: Silicon Carbide crystal (left) and examples of applications (right three), including a SiC abrasive wool, a SiC rotor in a brake disc, and a SiC MOFSET.

Experimental

XRD is the process in which a beam of X-rays are shot into a crystalline sample and the pattern and intensity of the diffracted X-Rays are measured. TGA-DSC is the process in which both heat flow and weight changes in a material are measured as a function of temperature and time in a controlled atmosphere. These two experiments together give insight to a material's composition.

The SiC sample provided by an industry source was plated uniformly on a Silicon wafer and placed into a Bruker D2 Phaser to perform an XRD scan from 10º to 80º 2theta at an increment of 0.01º (Fig. 2). A 12 mg sample of SiC was put into a TGA-DSC crucible and placed into a Setaram labSys Evo TGA-DSC and scanned from 30ºC to 1200ºC in an air atmosphere (Fig. 2).

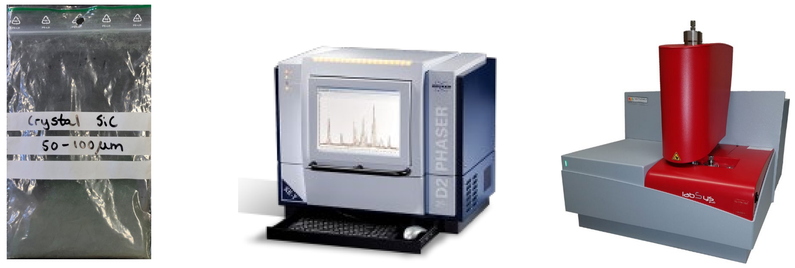

Figure 2: Industrially sourced SiC sample (left), D2 Bruker Phaser (middle) and Setaram labSys Evo TGA-DSC (right).

Results - Characterization

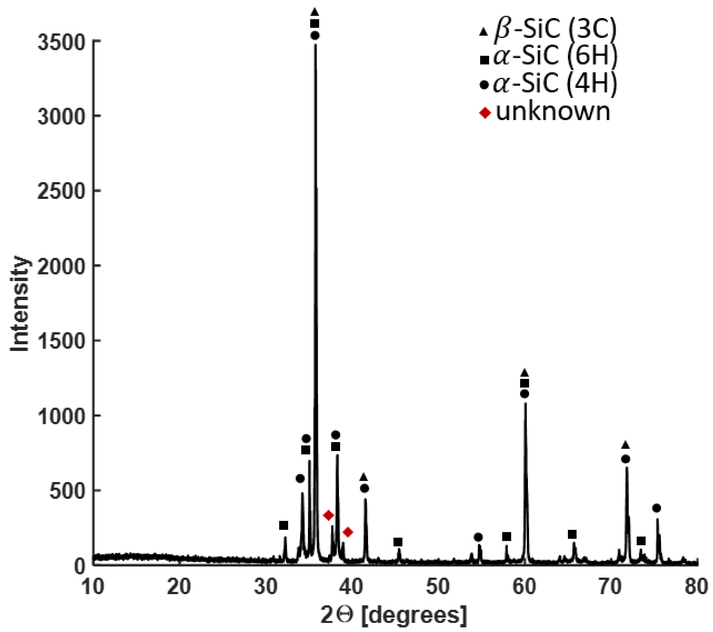

Figure 3: XRD scan of SiC sample showing intensity of diffracted X-Rays from 10º to 80º 2theta. Different SiC polymorphs were identified to be present within the sample. 3C β-SiC is shown as a black triangle, 6H α-SiC is shown as a black square, 4H α-SiC is shown as a black circle, and unidentified peaks are shown as a red diamond.

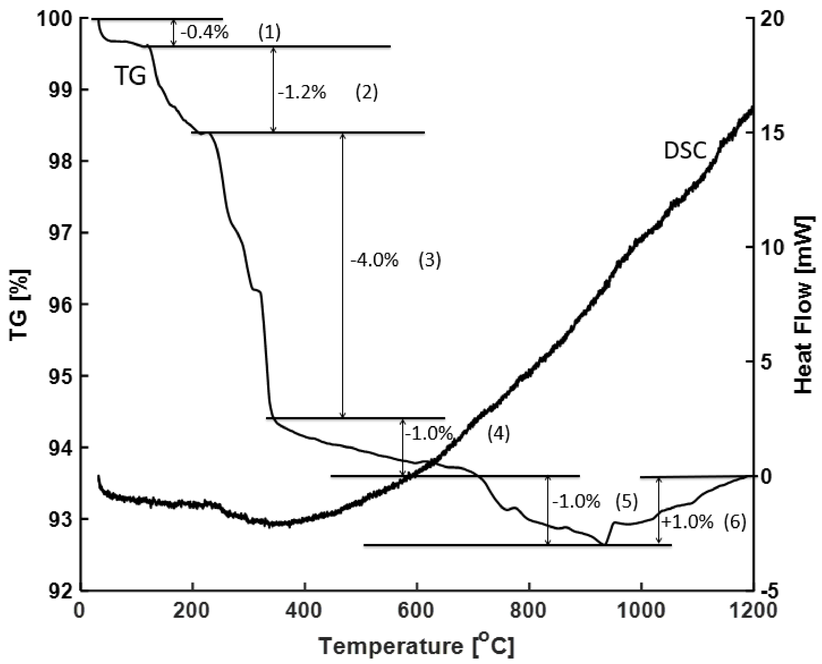

Figure 4: TGA-DSC scan of the SiC from 30ºC to 1200ºC in an air atmosphere with sections of mass changes labeled 1-6.

Discussion

Each peak present in the XRD scan was identified using previous SiC reference scans[5,6]. From this, three main SiC polymorphs are thought to be present within the sample: 3C, 6H, and 4H SiC. These three polymorphs differ in their crystal structure[7] (Fig. 5). Although different in crystal structure, the difference in energy states between each SiC polymorph is thought to be small enough to not affect calorimetric experiments. However, two peaks labeled as red diamonds were not matched with any SiC reference peaks. It is thought that the sample contains some substance other than SiC, which could greatly affect calorimetric experiments.

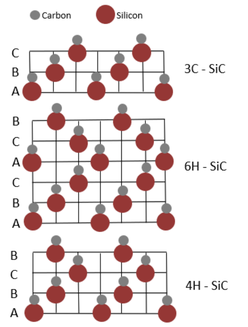

Figure 5: Stacking sequence of the 3C (top), 6H (middle) and 4H (bottom) SiC polymorphs.

The TGA-DSC scan shows that the SiC sample changes mass in many steps over, rather than gradually. In the first step, only 0.4% of mass is lost, which suggests that it is the evaporation of surface water. In the next two steps, mass is lost more suddenly and in greater magnitude, suggesting an evaporation of bulk water present inside the SiC sample. The fourth and fifth step occur over a larger temperature range, and lose less mass than before, suggesting a decomposition, possibly related to the impurity the XRD scan suggested was present in the sample. Finally, the last step is an oxidation on the sample's surface, due to the air atmosphere and high temperature.

Conclusion

XRD and TGA-DSC experiments were able to determine presence of multiple SiC polymorphs, water, and an unknown impurity in the industrially sourced SiC sample. Given that the impurity has an unknown energy state, performing calorimetry on this SiC sample would provide flawed results and give incorrect information about SiC's thermodynamic properties.

Moving forward, a new sample must be found and characterized, and then calorimetric experiments can be performed to develop an understanding of SiC's thermodynamic properties.

Acknowledgements

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