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# Experimental measurement of Young's modulus from a single crystalline cementite

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Pure Fe–C pearlite was heat-treated and selectively etched to extract [0 0 1]- and [1 0 0]-oriented single crystalline cementite sheets. The elastic properties of the shaped cementite were measured in a simple, in situ bending test system set up inside the scanning electron microscope using a micronewton-range force sensor. The Young's modulus experimentally measured from a single crystal sheet was lower than the value obtained from theoretical calculation.


*Keywords:* Cementite; Pearlitic steels; Bending test; Elastic behavior; Scanning electron microscopy

Pearlitic steel, which has a lamellar structure with alternating layers of ferrite and cementite, is one of the most commonly used structural materials in the industry. Cementite is one of the most important phases in steel performance and much research has been conducted on its mechanical, electrical and thermal characteristics [1–6]. The mechanical properties of pearlitic steel can be accurately predicted if the proper elastic constants of the cementite and ferrite phases are known. Experimental attempts were made to obtain the Young's modulus of the cementite from polycrystalline bulk cementite [5] and the multi-phase cementite [7–9] prepared through carbon replica [2], deep etching [4] and thin-film deposition [3,6]. The alternating nature of cementite sheet and ferrite shows that it is good to have the elastic properties of a single crystal cementite. Considering the anisotropy, most of the studies so far have used the polycrystalline phase due to the difficulty in preparing a single crystalline cementite. On the other hand, the cementite's elastic modulus theoretical calculations are widely available from many research groups [10–12].

In order to measure the mechanical properties from a single crystalline cementite, three major experimental hurdles need to be overcome. First, single crystalline cementite sheets with a specific crystallographic orientation in the lamellar structure of pearlitic steel should be extracted. Second, the geometrical arrangement of the extracted cementite sheets must pass a bending test that considers the anisotropic elastic constants of a single crystalline cementite. Finally, the bending test should have stress quantification on a small scale. In this study, we measured the Young's modulus of a single crystalline cementite along two different orientations, in the [1 0 0] and [0 0 1] directions, and compared the results with the theoretically calculated value from the reported elastic constants provided by the first-principles calculation.

Iron and carbon powders (99.9% pure) were mixed and melted to form the Fe–C target alloy in an induction heating system. The carbon content was a little less than

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0.8% of the ferrite and pearlite mixed microstructure. The steel sample was heat-treated for 60 min at 1200 °C and then slowly cooled to room temperature in order to homogenize the pearlitic microstructure that form the target lamella with a thickness of 100–200 nm. The surface was polished by etching in a 10% nital etching solution for 30 min to dissolve the ferrite phase. Cementite sheets were obtained by sonication of the etched surface immersed in ethyl alcohol. This was followed by spraying on a lacey carbon film supported by a Cu mesh grid.

Figure 1(a) shows the microstructure of the sample after the nital etching, the ferrite and cementite phases being visible as dark and light contrast, respectively. Figure 1(b) shows a scanning electron microscope (SEM) image of cementite sheets collected on the lacey carbon grid extracted from the etched sample. Hundreds of cementite sheets were examined and marked in a transmission electron microscope (TEM) to locate the cementite with the correct crystallographic orientation and size for the bending test.

The selection was done by using the TEM and SEM to find large sheets before taking diffraction patterns of individual cementite sheets to find and mark sheets with [010] orientation. A typical TEM bright-field (BF) image and selected area diffraction pattern (SADP) are shown in Figure 1(c) and (d), respectively. Holes were commonly observed in the cementite sheet, which further limited the choice of sheets used to make cantilever samples for the bending test. The holes in the cementite sheets are formed by the instability of the growth tip and local carbon distribution during the growth of the cementite phase from the austenite [13,14]. Most sheets had uniform thickness, which were judged, from their thickness and bending contours, to be suitable for the cantilever sample. Once the proper sheet size was selected, it was shaped and fixed for the cantilever beam-type bending test using a dual-beam focused ion beam (FIB) (FEI Quanta 3D and Nova).

In order to protect the cementite sheet from contamination and damage from the gallium ion beam, the FIB lift-out processing was carried out by the electron beam at all steps except during the fixing step at one end of the selected cementite sample. The Ga beam was used to deposit Pt to fix one end and shape the rectangular cantilever beam. Figure 1(e) and (g) shows TEM BF images of the cantilever beam-type cementite sheets ready for the bending test. The crystal orientation of the mounted cementite sheets was reconfirmed before the bending test, as shown in the typical cases in Figure 1(f) and (g), where the sheets were oriented in the (010) plane along the [100] and [001] directions, respectively. The longitudinal direction of the single crystal sheet was carefully chosen based on the previously reported elastic constants of cementite [15].

The home-made in situ bending test system was set up inside the SEM (JEOL JSM-6390). Shown in Figure 2(a), it consisted of three picomotor-driven actuators (Newport 8353-V) and *XYZ*-axis linear manipulating stages with a mounted force-sensing probe (FemtoTools FT-160). The loading force applied to the cementite sheet was measured by the force-sensing probe at an acquisition rate of 10 Hz, with a measurable minimum force of 0.5 μN. The force-sensing probe tip was created by using the FIB to make the point touch one side of the rectangular cementite sheet. The force sensor was calibrated using a Kleindiek calibration spring and a Nanosensors atomic force microscope tip prior to taking actual measurements. Figure 2(b) displays the schematic of the bending test, where the displaced sensing probe was held for 30 s at maximum displacement to stabilize the measured peak force before returning to zero displacement. This was necessary to confirm that the reading from the force sensor was fully stabilized without any changes over time. The force sensor's response was recorded in real time while observing the bending sequence in the test. The bending test was per-

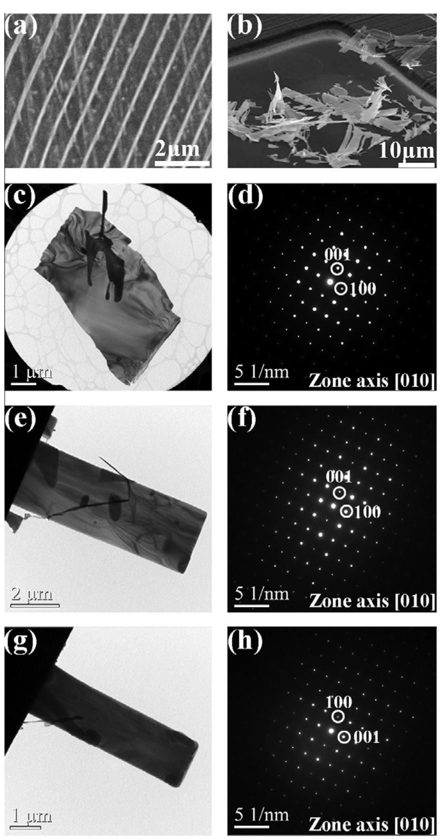


**Figure 1.** (a) SEM image of the pearlitic microstructure of a nital-etched sample. (b) SEM image of extracted cementite sheets on a lacy carbon film grid. (c) TEM BF image and (d) SADP of an individually extracted cementite sheet. (e) TEM BF image and (f) SADP of a cementite sheet along the [100] orientation with a [010] zone axis. (g, h) Cementite sheet along the [001] orientation.

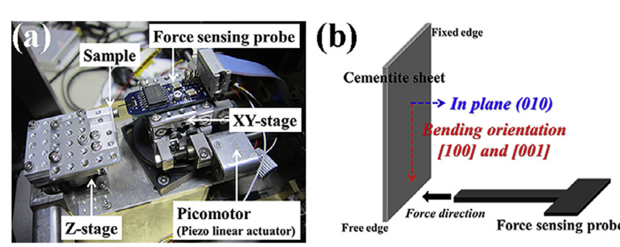


**Figure 2.** (a) Home-made in situ bending test system set up inside an SEM. (b) Schematic configuration of sensing the displacement and force for the bending test.