converted by cooling slowly from 700° to 900°C, Baer *et al.* (*8*) observed a decrease in resistivity on heating through the transition point. A decrease in resistivity was also observed by Junod (*2*). To explain their results, Baer *et al.* (*8*) have proposed two modifications of the low temperature phase. At atmospheric pressure the sign of the temperature coefficient of resistivity is negative for α-Ag_2Se and positive for β-Ag_2Se. This change in sign is observed for pressures up to about 15 kb, but at higher pressures the temperature coefficient is negative for both phases (Fig. 1). At most pressures the $\beta \rightarrow \alpha$ transition on cooling occurred 15° to 25°C lower than did the $\alpha \rightarrow \beta$ transition on heating.

Transition temperatures (obtained from graphs of resistance plotted against temperature, as temperature increases) are plotted against pressure in Fig. 2. For each pressure the transition temperature was taken as the temperature at which the resistance began to rise steeply. Roy *et al.* (*9*) observed an increase of 8.0°C in the transition temperature at 1115 atm with differential thermal analysis (DTA). As shown in Fig. 2, this point falls on the smooth curve connecting my data. The increase in transition temperature with pressure is consistent with crystallographic evidence that β-Ag_2Se (*2*) is less dense than α-Ag_2Se (*1, 3, 4*). [The face-centered tetragonal α-phase proposed by Junod (*2*) had lattice parameters which gave a molar volume which is inconsistent with the experimental data.]

The heat of transformation (ΔH_t) has been calculated from the pressure data by applying the thermodynamic relation $(dT/dp) = T\Delta V_m/\Delta H$. The initial slope of the curve of the transition temperature plotted against pressure from the data of Roy (*9*) is $dT/dp = 7.0$°C per kilobar. The transition temperature is 406°K. The molar volumes (V_m) of the α and β phases calculated from lattice parameter data are 35.57 cm^3/mol (*3*) and 37.36 cm^3/mol (*2*), respectively. The ΔH_t calculated from these values is 2.5 kcal/mol. Roy *et al.* (*9*) obtained $\Delta H_t = 1.678$ kcal/mol by using a molar volume for the low-temperature phase calculated from their measured density of 8.14 g/cm^3. The molar volume used in my calculation corresponds to a density of 8.29 g/cm^3.

Since the present calculated ΔH_t did not agree with reported values (1.68, 1.61 kcal/mol) determined from heat capacity measurements (*8, 10*), a calorimetric measurement of ΔH_t was made with a Perkin-Elmer Model DSC-1 differential scanning calorimeter (*11*). In this apparatus the sample and a standard are sealed in aluminum foil, heated or cooled at a constant rate, and the differential power necessary to keep the sample and standard temperatures equal throughout the run are recorded and integrated. For the measurements on Ag_2Se the calorimeter was calibrated by melting-freezing runs on high purity indium metal ($T_m = 156.6$°C, $\Delta H_m = 0.78 \pm 0.02$ kcal/g-atm). The $\alpha \rightarrow \beta$ transition on heating began at 132.5°C and the $\beta \rightarrow \alpha$ transition on cooling began at 113°C, in good agreement with the temperatures observed in the resistivity measurement. The ΔH_t for Ag_2Se was 2.19 kcal/mol.

In calculating the ΔH_t from pressure data the values of $V^{\alpha}{}_m$ and $V^{\beta}{}_m$ were obtained from lattice parameters measured at 298° and 443°K, respectively. Therefore, the value of ΔV_m used in the calculation is greater than the actual value at the transition temperature. Use of the value of ΔV_m at 406°K would reduce ΔH_t and improve its agreement with the result obtained calorimetrically. Roy *et al.* (*9*) made such a correction by measuring the increase in lattice parameter of the cubic high temperature phase with increasing temperature and by assuming the same expansion coefficient for the low temperature phase. However, they gave no data which could be used to correct the present calculations.

An unpublished (*12*) DTA resulting in $\Delta H_t = 2.36 \pm 0.59$ agrees better with the ΔH_t values obtained in this study than with the values from heat capacity measurements. The discrepancy between the heat capacity results and those obtained by other methods may be due to the existence of the two modifications of the low temperature phase proposed by Baer *et al.* (*8*). This difference of about 0.7 kcal/mol may be heat of ordering which they discussed, but for which they assigned no value.

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Screw Dislocations in Graphite

Abstract. *Graphite contains varying concentrations of screw dislocations whose Burgers vector parallels the* c *axis. Single crystals of natural graphite contain very few such dislocations; furthermore, their Burgers vector always exceeds 450 angstroms. Pyrolytic graphites annealed above 3000°C contain abundant screw dislocations, ranging from 10^6 to 5×10^8 per square centimeter in two different samples prepared by somewhat different methods. The Burgers vectors of these screws are predominantly 3.35 angstroms.*

A decoration technique has been developed (*1, 2*) which can render individual lattice vacancies in graphite visible for electron microscopy. The method consists of cleaving the crystal to a thickness of a few hundred angstrom units, etching in a gas (usually a mixture of O_2 and Cl_2) which expands surface vacancies to any desired diameter without increasing their depth, and finally decorating with minute amounts of gold. This method reveals other lattice defects besides vacancies, and in particular can be used to identify screw dislocations by the extra surface step which originates on the dislocation (*1*). I now report that the screw dislocations can actually be "unravelled" by suitable etchants like carbon dioxide which react faster at linear than highly curved surface steps. After the core of the screw has been etched away, the remaining

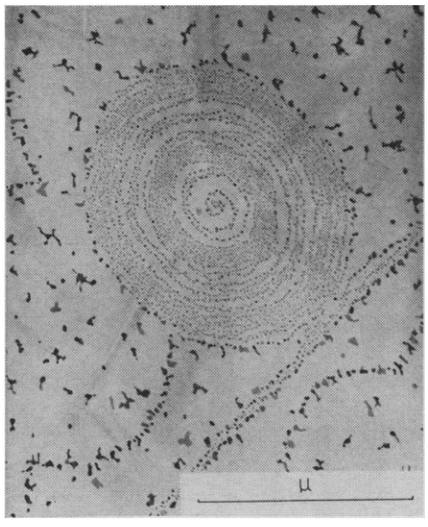

Fig. 1. Screw dislocation in "G.E." pyrographite, etched 46 minutes, CO_2, 1150°C.

surface steps are oxidized at an increasing rate, and thus the screw pattern is magnified (Fig. 1 and cover).

The magnitude of the Burgers vector (equal to pitch) of the screw can be determined by any procedure which measures the height of the step originating on the dislocation. For large steps which could, of course, be measured only by replication techniques, conventional shadowing techniques had been used (*3*). For monatomic steps, that is, steps which are only one atom layer high, the step height can most readily be demonstrated by creating additional surface steps known to be only one atom layer high, and etching the surface until the various steps merge. Monolayer steps can be created at will by any chemical or physical procedure which creates single vacancies. The most convenient one for the present problem was the creation of single surface vacancies by chemical attack with ozone. Detailed calibrations (*4*) had established that at 40°C a gas mixture of 10 percent ozone in oxygen created 10^{-6} surface vacancies per atom per minute and only negligible amounts of multilayer vacancies during the first hour of reaction.

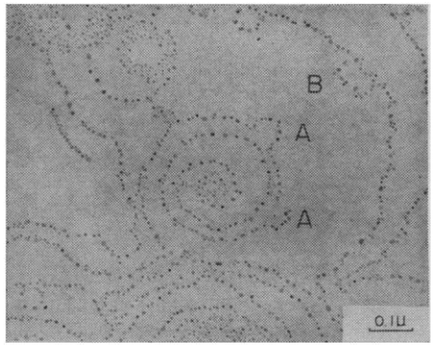

Fig. 2. Determination of Burgers vector. "French" pyrographite etched 24 minutes, CO_2, 1150°C; 1 minute, O_3, 40°C; 10 minutes, Cl_2–O_2, 650°C. *A*, Merged surface steps of dislocation and of loop. *B*, Nonmerging steps, indicating multilayer defect.

The cleaved crystals containing screw dislocations were, therefore, first etched in carbon dioxide to develop well-spaced screw steps, then treated with ozone to produce single surface vacancies, and finally etched briefly in a mixture of O_2 and Cl_2 to expand the vacancies into surface depressions large enough to guarantee frequent intersections with screw steps. An example of four such intersections is shown in Fig. 2. Two of the intersections, designated *A*, show true merging without any additional step between the loop and the screw. They demonstrate that the screw of Fig. 2 consists of a monatomic surface step, that is, a step 3.35 Å high, and that the *c*-component of the Burgers vector of the screw dislocation at the center of the screw is 3.35 Å. The intersection designated *B* shows an example of a multilayer step, which is, however, not part of a screw dislocation.

These methods have been applied thus far to three types of graphite. Natural graphite was obtained from the Ticonderoga area of northern New York State. This graphite is found both in igneous intrusion and in Precambrian limestone, in the form of crystals which are often of great perfection. Literally thousands of these have by now been examined by our group without revealing a single example of a monatomic screw dislocation. We estimate that we have searched at least 1 cm^2 so that an upper concentration limit of one screw per square centimeter is indicated. This estimate neglects the possible occurrence of isolated patches of screws, which could obstruct easy cleavage of the crystal and thus escape detection; nonetheless, patchy cleavage was rarely observed. The same crystals contain, however, an appreciable concentration of large screw dislocations (*3*) whose pitch is never less than 450 Å high, and which generate occasional spiral surface growth patterns (*3, 5*).

Pyrolytic graphite (*6*) had been recrystallized by heating above 3100°C under pressure. This material contained at least 5×10^8 screw dislocations, about equally left and right handed. Nearly all the screws which could be examined in detail had a pitch of one atom layer (Fig. 2). About five percent of the screws were very short, terminating within two or three layers. The concentration of screws may have exceeded $5 \times 10^8/cm^2$ because elongated crowded patches rendered precise determinations difficult.

A sample of pyrolytic graphite (*7*), which had been deposited at high supersaturation of carbon and subsequently had been heated in a graphite capsule at 3600°C, contained 10^6 screw dislocations per square centimeter.

Both samples of pyrographite were difficult to cleave and contained numerous defects other than the screw dislocations. The size and density distribution and the behavior of the screws on further annealing is still uncertain.

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Electron Microprobe Analysis of Oxygen in an Iron Meteorite

Abstract. *A quantitative analysis of oxygen in the iron-nickel matrix of the Santa Catherina iron meteorite has been accomplished by using an electron microprobe with dispersive x-ray optics and by applying theoretically calculated corrections to the observed intensity ratios. Microvolume analyses and two-dimensional scanning images in OKα, FeLα, and NiLα x-radiation show that the high nickel phase of the meteorite has been oxidized while the low nickel phase has remained unoxidized.*

The Santa Catherina iron meteorite is a nickel-rich ataxite with an overall nickel content of 36.14 percent by weight (*1*). Considerable importance has been attached to this meteorite because it appears to have originally consisted of two coexisting iron-nickel alloys of the high nickel γ-phase (*2*) which have not been found in other iron meteorites or man-made alloys. Polished sections