4. Delamination of Pressed Specimens

It has been observed that many of the MgO specimens have been severely laminated, giving thin disks from 0.030 to 0.125-inch thick upon removal of the nickel heater sleeve. It is believed that this delamination resulted from elastic recovery during the unloading from the high-pressure condition. The lower elastic limit of the nickel sleeve resulted in its being under axial compression and the MgO under axial tension after the release of pressure. An approximate calculation of the tension on the MgO may be made. Before unloading, the system is in equilibrium at some pressure P_{max}. Upon unloading, with no restraints, the nickel sleeve will expand by an amount

$$\Delta \ln L = \frac{1}{E} \frac{\Delta F}{A} \qquad (4)$$

where:

L is the length
E is Young's modulus
ΔF is the change in force on the sleeve
A is the cross-sectional area of the sleeve.

Using the subscript 1 for MgO and 2 for the nickel sleeve we have, since upon unloading both the sleeve and MgO expand by the same amount,

$$\Delta \ln L = \frac{1}{E} \frac{\Delta F_1}{A_1} = \frac{1}{E_2} \frac{\Delta F_2}{A_2} \qquad (5)$$

but,

$$\frac{\Delta F_1}{A_1} = P_{max} - P_{1, final} \qquad P_{max} - P_{1, final} \qquad (a) \qquad (6)$$

and,

$$\frac{\Delta F_2}{A_2} = P_{max} - P_{2, final} \qquad P_{max} - P_{1, final} \qquad (b)$$

However, after unloading, the force on the nickel must be equal and opposite to that on the MgO so that

$$A_1 P_{1, final} = -A_2 , P_{2, final} \qquad (7)$$

Substituting Equations (6) and (7) into (5) we obtain

$$P_{1, final} = P_{max} \frac{(E_2 - E_1)}{(E_2 + \frac{A_1}{A_2} E_1} \qquad (8)$$

$P_{1, final}$ will be negative (that is the MgO will be under tension) if E_1 is greater than E_2. Typical values of E are 45 x 10^6 psi for MgO[21] and 30 x 10^6 psi for nickel[22].

At the end of the run, the diameter of the sample is about 0.375 inch and the nickel sleeve has a thickness of at least 0.010 inch. The area then is given by

$$\frac{A_1}{A_2} = \frac{\pi (0.187)^2}{2\pi (0.187)(0.010)} = 9.3 . \qquad (9)$$

Substituting these values and P_{max} = 250, 000 psi into Equation (5) we obtain

$$\begin{aligned} P_{1, final} &= 250{,}000 \; \frac{(30 - 45)}{30 + (9 \times 45)} \\ &= \frac{15}{435} \times 250{,}000 \qquad (10) \\ &= -8600 \text{ psi} \end{aligned}$$

The ultimate tensile strength of a typical dense fine-grain MgO is of the order of 15, 000 to 18, 000 psi.[23] Thus this simple calculation shows a tensile force of about one-half of the strength of the material. The nickel tube deforms severely during the pressing operation, developing transverse wrinkles which (1) provide good contact with the MgO for the transmission of axial forces, (2) possibly introduce stress risers in the MgO at these wrinkles, and (3) may, by changing the area ratio, lead to higher local stresses.

If this model represents correctly the reason for delamination of the specimens, such delaminations may be avoided by:

a. choosing a sleeve material with a higher Young's modulus,

b. using a thinner or weaker sleeve, and/or

c. controlling the delamination by the introduction of shims.

Using the second suggestion a graphite sleeve was used as a heater. The resultant load applied to this graphite sleeve compresses the sleeve as well as the sample without breaking the electrical current. The collapsing of the tube did not increase the internal stresses as was the case for the nickel tubes. This was later proven when large intact pieces were obtained using the graphite sleeve.