

approximately 1000° C. Sample 21A did not show any grains which could be measured. "Pores", however, were observed with an average size of approximately 500A, that is, of the same order as the starting powder. The shape of the "pores" suggested to the microscopist that they were pull-outs rather than real pores. Sample 20 showed better definition of the grains with some being extracted with the replica. The hardness measurements were 1187 and 1095 KHN (average of 5 each) with a 100 gram load. Electron micrographs of these two specimens are shown in Figure 8 and 9. A thin chip of sample 21 was chemically thinned for transmission electron microscopy. This is shown in Figures 10a & b at 240,000X showing an average grain size of approximately 500A.

The early experiments with MgO, runs 10B and 12B had shown that essentially complete densification would be achieved with a minimum of grain growth and the development of high hardness. A study of the processing variables was made in runs 45C and 53C to 77C. The relation between temperature and time under pressure as independent variables and grain size and hardness as dependent variables was investigated. In this series, the pressure was kept constant at 110,000 psi (7.5 kilobars). In Figure 11, the grain size as determined by X-ray line broadening is plotted against the temperature of the run. Above about 900° C considerable grain growth is evident although run 74C shows no appreciable grain growth, even at 1025° C. There is very little effect of time; most of the grain growth appears to develop within the first 10 minutes at temperature.

The results of Knoop hardness measurements are shown in Figure 12. Although the results are not conclusive, there is a definite evidence of a maximum at about 900° C. Above this temperature grain growth occurs, below this temperature insufficient densification is obtained. In Figure 13 the hardness is plotted against the grain size. The considerable scatter at small grain size reflects the differing degrees of densification in the various samples. Thus, the high values represent those specimens which had density without appreciable grain growth while the low values represent those specimens run at lower temperature which had not densified as much.

The extremely high hardness observed in these preparations suggested possible connections between hardness and optical properties, as suggested by the contract monitor. To evaluate this, a number of runs were made to prepare specimens exhibiting the high hardness observed in these experiments and these specimens were submitted to AFCRL for further study. They are listed in Table VII.

SAMPLE # 20

GRAIN SIZE = $0.26\ \mu$



63TI5

(A)

28,000 X



63TI8

(B)

55,000 X

Figure 8 ELECTRON MICROGRAPHS OF HIGH DENSITY MgO

SAMPLE #21



63RI0

55,000 X

Figure 9 ELECTRON MICROGRAPHS OF HIGH DENSITY MgO