THE DEVELOPMENT OF FERROMAGNETIC SPINELS
FOR OPTICAL ISOLATION AT 10.6 μm

Progress Report

MASTER

December 1, 1977 to February 28, 1978

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ABSTRACT

During the period December 1, 1977 to February 28, 1978 emphasis has been placed on preparing pure powders of CdCr_2Se_4 and determining the parameters necessary to hot press these into windows with low absorption at $10.6\text{ }\mu\text{m}$. Two methods of preparation, namely growth of small single crystals from a liquid phase and the Kodak technique of coprecipitation of hydroxides and subsequent treatment with H_2Se have yielded samples which have an absorption coefficient of $\sim 100\text{ cm}^{-1}$ at $10.6\text{ }\mu\text{m}$. The samples contain Cr_2O_3 as a major impurity. Scattering due to either pores or a second phase also limits transmission. Procedures for removing Cr_2O_3 and decreasing porosity are discussed.

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I. Current Status

During this period work has continued on the preparation and hot pressing of CdCr_2Se_4 . Three different methods of preparation have now been used. These are the growth of small single crystals through a liquid phase reaction of CdSe , CrCl_3 and Cd ; direct reaction of CdSe_4 and Cr_2Se_3 ; and co-precipitation of Cd and Cr hydroxides with subsequent conversion into the spinel accomplished by treatment with H_2Se . This latter method of preparation was carried out by E. Carnall and D. Pearlman at the Eastman Kodak Research Laboratories.

All of these materials have been examined by X-ray analysis and second phase contamination found to be less than .5%, the minimum detectable by this technique. They have been hot pressed and the absorption spectrum measured in the infrared to determine the absorption coefficient at 10.6 μm and to identify optically active impurities or phases. In addition the density of hot pressed samples has been measured to monitor the porosity of the materials.

The hot pressed samples prepared from powdered crystalline material and from CdCr_2Se_4 formed from precipitated hydroxides had absorption coefficients of $\sim 100 \text{ cm}^{-1}$ at

10.6 μm . In thin sections these were transparent enough to permit infrared absorption measurements. The transparent samples all showed absorption bands due to Cr_2O_3 and scattering related either to a second phase or residual pores on grain boundaries. The density of the hot pressed materials was found to be within .5% of the theoretically obtainable value. This result shows that as far as obtaining good densities is concerned, our hot pressing techniques are at least equal to those found to produce good samples of CdCr_2S_4 .

In addition to the above work, a project to prepare a hot pressed room temperature ferro or ferri magnetic spinel was initiated. $\text{CdCr}_{2-x}\text{Fe}_x\text{Se}_4$ is being prepared to see if enough Fe^{+++} can be substituted for Cr^{+++} to raise the Curie temperature above room temperature without a phase change to the metallic NiAs structure. This work is still in its preliminary phases.

Studies to determine the laser damage threshold for CdCr_2Se_4 at 10.6 μm are continuing. Before reporting such a threshold, an attempt is being made to develop a meaningful criterion for damage and to establish a reliable technique for monitoring both temporal and spatial variations in the power density of our pulsed CO_2 laser. No significant difference between the damage threshold for CdCr_2Se_4 and CdCr_2S_4 has as yet been found.

Mr. Robert Freese and the Principle Investigator presented a paper at the CLEOS conference in San Diego during this quarter. Also, a paper was presented at the topical meeting on inertial confinement by the same authors. The presentations were entitled "Faraday Effect Optical Isolation at 10.6 μm ," and "Hot Pressed CdCr_2S_4 for Optical Isolation 10.6 μm ."

II. Plans for the Next Quarter

During the next quarter we will refine powders of CdCr_2Se_4 produced by the techniques described above and determine the hot pressing parameters needed to minimize absorption at 10.6 μm .

Purification of the starting powders will be attempted in two ways. Pure H_2 and excess selenium will be used to reduce Cr_2O_3 and incorporate the Cr^{+++} into the spinel structure, and as an alternate technique treatment with CCl_4 will be used to convert second phase Cr_2O_3 to CrCl_3 and CO_2 , following a well known method for preparing anhydrous CrCl_3 . The infrared absorption of CrCl_3 is at much longer wavelengths than Cr_2O_3 and hence may have much less effect on the absorption at 10.6 μm . Also, CrCl_3 is slightly soluble in hot H_2O , and hence can be removed if it persists as a second phase.

The influence of hot pressing parameters on absorption in CdCr_2Se_4 will be investigated. It is known that the

optical absorption of hot pressed CdCr_2S_4 at $10.6 \mu\text{m}$ is most strongly influenced by the temperature of pressing, rather than pressure or pressing time. Therefore we plan to initially vary the temperature of hot pressing to minimize optical absorption in CdCr_2Se_4 . Other parameters will then be optimized to further refine the optical properties. In connection with these studies, a sensitive technique for measuring the density of hot pressed samples is being developed to more accurately monitor porosity and scattering.

It is hoped that a damage threshold for CdCr_2Se_4 will be established during the next quarter. Also, work on a possible room temperature isolator material will continue.

The Principle Investigator will spend 30% of his time on this contract during the next quarter.