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EXAMINATION OF TRANSMISSION OF COPPER SULFIDE LAYERS FORMED ON CADMIUM SULFIDE USING CUPROUS CHLORIDE SOLUTIONS

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16. Abstract

Optical transmission studies were made of copper sulfide barriers formed on CdS films and compared with transmission curves of known copper sulfide. The effect of halide ion concentration in the solution was examined. To form a cuprous sulfide barrier, the cuprous chloride solution must apparently contain a sufficient concentration of a complex ion such as CuX_2^- (X = Cl or Br). A barrier of mixed copper sulfides formed when this contentration was too low. The effects of bath temperature and dip time are very critical to barrier thickness. Prolonged washing of the cell after barrier formation converts some of the barrier to other forms of copper sulfide.

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SUMMARY

Optical transmission studies were made of the formation of copper sulfide barriers on cadmium sulfide thin films by a chemical displacement reaction in cuprous chloride solutions. The effects of copper ion concentration, pH, dip time, bath temperature, and subsequent washing of the cell were studied.

To obtain the cuprous sulfide barrier, the cuprous chloride bath had to contain a sufficient concentration of a complex copper ion (e.g., $CuCl_2^-$, $CuBr_2^-$). This concentration can readily be achieved by adding a sufficient quantity of sodium chloride, potassium chloride, sodium bromide, or potassium bromide. When the complex ion concentration is too low, a barrier forms that probably contains cuprous sulfide plus other copper sulfides or copper. Sodium and potassium iodide gave similar results, but they could be used only over a limited concentration range.

The main effect of other bath variables such as temperature and dip time is to change barrier thickness. These variables must be held fairly constant to ensure uniform results.

The cuprous sulfide barrier was converted to other forms of copper sulfide by extensive washing after barrier formation.

INTRODUCTION

The cadmium sulfide (CdS) solar cell consists of a barrier of copper sulfide on a single crystal, ceramic plate, or film of CdS. The copper sulfide barriers have been applied in several ways. Chamberlin and Skarman (ref. 1) sprayed a barrier of copper sulfide onto CdS films. Shitaya and Sato (ref. 2) and Nakayama (ref. 3) have produced barriers on single crystals and ceramic plates by electroplating them in a dilute aqueous solution of $CuSO_4$. Several other techniques including evaporated barriers of Cu_2S were tried in reference 4, but these were unsuccessful. Shirland (ref. 5) reports that very satisfactory Cu_2S barriers are obtained by dipping the CdS into a heated, aqueous solution containing cuprous ions, generally a bath of cuprous chloride (CuCl).

Although the CuCl bath process has been used extensively and proved successful, the exact mechanism of the overall reaction and the effect of individual variables are not well understood. This study was therefore undertaken to examine this process.

Different analytical techniques for examining the barrier were tried. Because the barrier is very thin (~3000 Å or 300 nm) standard techniques, such as x-ray and chemical analysis, are not suitable. Optical transmission measurements offered a satisfactory and convenient way of following changes in barrier composition.

Optical transmission measurements revealed the effects of bath variables such as cuprous ion concentration, pH, dip time, bath temperature, and washing after barrier formation. From these results, the conditions for obtaining uniform barriers were defined.

PROCEDURE

Cadmium Sulfide Films

The CdS films used in this study were evaporated on glass substrates. Presintered General Electric luminescent grade CdS was used as the starting material. The CdS was sintered in argon for 64 hours at 800° C and then for 8 hours at 1200° C. The CdS was evaporated for 13 minutes. The resulting thickness was 34.6 micrometers which indicates an evaporation rate of 2.66 micrometers per minute. Substrate temperature was 228° C. Resulting carrier concentration in the films as determined by Hall measurements was 1.6×10^{17} e/cm^3.

Cuprous Chloride Bath

The CuCl solution was formed by adding 15 grams of CuCl to 1 liter of deionized water. This amount of CuCl provided an excess above solubility. The CuCl was washed with 6 normal hydrochloric acid (HCl) to remove cupric ions and was dried in a vacuum oven before use. Chemical analysis indicated an initial purity of 96 to 98 percent. The water was preboiled and kept under a nitrogen atmosphere to prevent oxidation of the cuprous ions. During dipping the acidic solution was stirred at a constant speed with a motor-driven stirrer.

Etching

In the manufacture of solar cell, the CdS film is generally etched in 3 to 1 solutions of HCl and H_2O or HBr and H_2O for a few seconds prior to dipping in the barrier solution. However, this etching, especially using HCl, greatly reduces the transmission of the CdS film. Therefore, only a few experiments were made with etched films in order to determine whether the barrier was influenced by etching.

Transmission Measurements

The effect of chemical and processing variables on barrier formation was observed by measuring the optical transmission with a Perkin-Elmer Model 350 spectrophotometer. The transmission of the films was measured before and after barrier formation. The transmission of the barrier was determined by dividing the transmission of the film after barrier formation by the transmission of the film before barrier formation. No corrections have been made for reflection losses or scattering effects.

RESULTS AND DISCUSSION

Forms of Copper Sulfide

Several forms of copper sulfide have been reported in the literature. The most common are Cu_2S, chalcocite; $Cu_{1.8}S$, digenite; $Cu_{1.96}S$, djurleite; and CuS, covellite. The transmission curves for Cu_2S, $Cu_{1.8}S$, and CuS are shown in figure 1 along with that for copper (Cu). No transmission curve is available for $Cu_{1.96}S$.

The curve for the 3000 Å (300 nm) film of Cu_2S was calculated from the absorption coefficients given in reference 6. For Cu_2S films thinner than approximately 5000 Å (500 nm), two band edges are observed: one at about 1.2 electron volt (1 μM) and another at 1.8 electron volt (0.7 μM). Thicknesses less than or equal to 5000 Å (500 nm) probably represent the thickness range of the barrier layers (ref. 6).

The $Cu_{1.8}S$ transmission curve is from reference 7. No thickness was given. The 3000 Å (300 nm) CuS and the 400^{o} Å (40 nm) Cu curves were obtained using evaporated films of CuS and Cu. The first band edge of Cu_2S is also reported by Marshall and Mitra (ref. 8) to be 1.2 electron volt (1.0 μM).

Effect of Cuprous Copper Ion Concentration and Bath Composition on Barrier Composition

The CuCl bath described earlier was heated to 90° C. An unetched CdS film was dipped for 5 seconds. The transmission curve of the CdS film before and after barrier application is shown in figure 2 along with the resulting transmission curve of the barrier. Etching the CdS film for 5 seconds in a 3 to 1 solution of HCl and H_2O before dipping produced no significant change in the barrier transmission curve.

A comparison of these data with the transmission curves in figure 1 suggests that the barrier does not correspond to any of these copper sulfides. No positive identification of this barrier has been made. One possible explanation is that the barrier may be a combination of Cu_2S and one or more of the other copper sulfides and/or Cu. This hypothesis was checked by measuring the transmission curves of a Cu film and a Cu_2S film together. The resulting curve was very similar to that shown in figure 2. The same results would probably be obtained by combining curves of Cu_2S with CuS or $Cu_{1.8}S$ although suitable films were not available for test.

CdS solar cells (on molybdenum substrates) made with this barrier were inefficient (<1 percent) when measured at air mass zero (AMO) intensity and 25° C. Thus, it appears that this barrier is not satisfactory for cell fabrication.

The cuprous ion (Cu^+) concentration present in this solution, which is limited by the solubility of CuCl, was about 1×10^{-3} moles per liter. This was calculated from the solubility constant of CuCl (ref. 9) as follows:

$$\begin{aligned} &CuCl \rightarrow Cu^+ + Cl^- \\ &K_{SP} = [Cu^+][Cl^-] = 1\times10^{-6} \end{aligned} \qquad (1)$$

Reference 10 indicates that in order to increase the solubility of CuCl, a soluble complex ion (e.g., $CuCl_2^-$, $CuCl_3^{=}$) must be formed. Adding Cl^- to achieve this effect could be accomplished by adding HCl. The effect on barrier transmission of adding HCl and thereby decreasing the pH of the bath from 4.73 to 3 is shown in figure 3. The barrier transmission is reduced, but the shape of the curve is still the same as in figure 2.

At a pH of 3, the chloride concentration is 1×10^{-3} mole per liter and the concentration of $CuCl_2^-$ was calculated to be 6.6×10^{-5} mole per liter based on the following relation (ref. 11):

$$\left.\begin{aligned} &CuCl + Cl^- \rightarrow CuCl_2^- \\ &K_{S_2} = \frac{[CuCl_2^-]}{[Cl^-]} = 6.6\times10^{-2} \end{aligned}\right\} \quad (2)$$

The cuprous (Cu^+) ion concentration was still about 1×10^{-3} mole per liter. Thus, the $CuCl_2^-$ plus the Cu^+ concentrations had not changed sufficiently to bring about a change in barrier composition. Although the Cl^- concentration could be increased by adding more HCl and thus lowering the pH further, lower pH values could lead to undesirable etching of the CdS film. Further, from the device viewpoint, Cusano (ref. 12) indicates that pH values lower than 3 should be avoided. Therefore, an increase in the concentration of the complex copper ion was obtained in another way. A pH of 3 was maintained for all of the following studies since this condition is comparable to most other reported studies.

References 13 to 15 indicate that the complex ion concentration can readily be increased by the addition of a salt. The results of the addition of sodium chloride (NaCl) are shown in figure 4. The addition of 0.167 gram per liter of NaCl had no effect, but 1.83 grams per liter changes the transmission curve to that more closely resembling Cu_2S. There are, however, some variations between this barrier and the Cu_2S film shown in figure 1. These variations may be due to the presence of small amounts of Cu or other copper sulfides. For discussion purposes we will hereafter refer to this barrier as Cu_2S.

When cells were made with this barrier, efficiencies of 5 percent and more were obtained (air mass zero and 25° C). These findings are in agreement with the results of reference 15. Thus, this appears to be a desirable barrier composition.

As shown in figure 4 the same transmission curve was obtained for concentrations of NaCl ranging from 1.83 to 15.2 grams per liter. The same range of concentrations of potassium chloride (KCl) was also examined with similar results. Table I shows the cuprous ion and the $CuCl_2^-$ concentrations for each of the NaCl concentrations used. Calculations of the concentrations of other complex ions were not made because of insufficient constant of formation data. The exceedingly high concentration of NaCl (200 g/liter) used in reference 15 was not found to be necessary to form the Cu_2S barrier.

Apparently, the Cu^+ ion is not effective in forming the Cu_2S barrier because at 1×10^{-3} mole per liter $[Cu^+]$, no Cu_2S was observed. At a comparable $CuCl_2^-$ ion concentration, the Cu_2S barrier formed. One possible explanation is that the charge on the ion plays a strong role in the reaction.

To determine whether etching the CdS film would affect this barrier, a study was made using 3 to 1 solutions of HCl and H_2O and HBr and H_2O as etchants. Hydrobromic acid and hydrochloric acid etching were reported to be equally effective in producing cells with good efficiency (ref. 16). No significant difference in barrier transmission could be detected with and without the HCl etch. The transmission of the CdS film and barrier was so low, however, that small differences could be easily masked. The HBr etched films, however, did transmit a sufficient amount of light to detect smaller changes. The results are shown in figure 5. The barriers are the same on etched and unetched CdS surfaces. Therefore, subsequent studies were made with unetched CdS films.

A similar study was made using sodium bromide (NaBr) and potassium bromide (KBr) instead of NaCl. The transmission of the barriers as a function of added NaBr is shown in figure 6. The pH of the bath was 3. Table I shows the Cu^+ and $CuBr_2^-$ concentrations. The value used for K_{S2} was 4.4×10^{-3} (ref. 11). With 33.3 grams per liter of NaBr (concentration of $CuBr_2^-$, 1.4×10^{-3} mole/liter), the Cu_2S barrier was obtained. To obtain the necessary concentration of the complex ion, about 33.3 grams per liter of NaBr are required, but with NaCl only 1.83 grams per liter were needed. This is a result of the order of magnitude lower stability of the complex $CuBr_2^-$ ion.

Both NaI and KI were also used as the added salt. The transmission of the barriers as a function of added NaI is shown in figure 7. The cuprous (Cu^+) ion and CuI_2^- ion concentrations are shown in table I. The value used for K_{S2} was 1×10^{-3} (ref. 11). About 2 grams per liter of NaI is needed to form the Cu_2S barrier. The concentration of CuI_2^- ion is only 1.34×10^{-5} mole per liter. This value is about two orders of magnitude lower than with the other salts. At a higher concentration of 15 grams per liter it was difficult to form a barrier. No detectable barrier formed in the 5-second dip. With a 12-second dip a very thin Cu_2S barrier was formed. Apparently, iodide ions act to dissolve Cu_2S, much like cyanide ions. If an iodide salt is used in the bath, only a limited range of concentrations can be used to form the Cu_2S barrier.

Effect of Dip Time

A study was made of the effect of dip time with and without a salt present. The pH of the bath was 3 and the bath temperature was 90° C.

The effect of changing the dip time from 3 to 25 seconds without the addition of a salt is shown in figure 8. At a dip time of 25 seconds, the barrier appears more like the Cu_2S barrier formed with a salt.

The effect of changing the dip time from <3 to 8 seconds with the addition of NaCl is shown in figure 9. Increasing the dip time decreases the barrier transmission. If a change in barrier thickness is required, it can readily be achieved by changing the dip time.

Effect of Bath Temperature

The effect of varying the bath temperature from 80° to 95° C is shown in figure 10. Dip time was 5 seconds at all temperatures, and NaCl was added. For the lower temperatures (80° and 85° C) the curves are very similar to that for the short dip (<3 sec) at 90° C in figure 9. (There thus appears to be some range over which time and temperature can be varied and yield the same result.) At the higher temperature the barrier transmission decreases sharply with increasing temperature. At 95° C the shape of the curve has changed somewhat; the barrier may no longer be Cu_2S. These results indicate that temperature must be controlled carefully to obtain reproduceable barriers.

Effect of Washing Cell After Barrier Formation

The Cu_2S barriers were washed immediately after formation by shaking them for about 30 seconds in a beaker of deionized water. This is the standard wash suggested in reference 16. Figure 11(a) shows the change in barrier transmission when the barriers are washed for 30 seconds and for 20 minutes in a beaker of still water. Figure 11(b) shows the results of washing the barriers in an ultrasonic cleaner for 5, 15, and 30 minutes.

Apparently, water causes the barrier to undergo some conversion process. Increased agitation as in the ultrasonic cleaner speeds up the conversion.

Evaporated films of Cu_2S, both on glass and on CdS films, were washed under the same conditions. They did not show this change in transmission. It would therefore appear that some significant difference exists between chemically formed barriers and evaporated Cu_2S films.

SUMMARY OF RESULTS

The following results were obtained from the study of the transmission curves of copper sulfide barriers formed on cadmium sulfide films. It was shown that, depending on the complex copper ion concentration, two kinds of barrier layers were formed. A barrier layer that strongly resembled cuprous sulfide was formed when the complex copper ion concentrations was increased sufficiently. These increases in complex copper ions were obtained by adding soluble halide salts (e.g., NaCl and NaBr) to the solution.

When the concentration of complex copper ions was too low, an unidentified barrier, probably consisting of mixed copper sulfides, was formed at dip times of about 5 seconds. When no additional salts were added to the bath, increasing the dip time to 25 seconds reduced the transmissions and also changed the composition of the barrier. When sodium chloride was present, increasing the dip time from <3 to 8 seconds decreased barrier transmission.

When NaI was used as the soluble halide salt, it was found that only a very limited concentration range could be used to obtain cuprous sulfide barriers.

The barrier composition results performed on unetched films were unaffected by etching the CdS film in either hydrobromic acid or hydrochloric acid before dipping. Raising the temperature of the bath from 80° to 95° C reduced the transmission of the resulting barrier. It also appeared that at 95° C some slight change in barrier composition had taken place.

Finally, extensive washing in deionized water causes the cuprous sulfide barrier layer to change composition. This change in composition could be accelerated by ultrasonic agitation during the washing.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 22, 1970,
120-33.

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TABLE I. - COMPLEX ION CONCENTRATION USED IN BATH

Salt	Concentration, g/liter	Ion concentration, mole/liter			Barrier
		$[X^-]$	$[Cu^+]$	$[CuX_2^-]$	
NaCl	0.17	0.0029	3.5×10^{-4}	1.9×10^{-4}	Not Cu_2S
	1.83	.032	3.2×10^{-5}	2.1×10^{-3}	Cu_2S
	6.83	.12	8.6×10^{-6}	7.7×10^{-3}	Cu_2S
	15.2	.26	3.9×10^{-6}	1.7×10^{-2}	Cu_2S
NaBr	0.17	0.0016	6.2×10^{-4}	7.2×10^{-6}	Not Cu_2S
	1.83	.018	5.6×10^{-5}	7.8×10^{-5}	Not Cu_2S
	6.83	.066	1.5×10^{-5}	2.9×10^{-4}	Not Cu_2S
	15.2	.15	6.8×10^{-6}	6.5×10^{-4}	Not Cu_2S
	33.3	.32	3.1×10^{-6}	1.4×10^{-3}	Cu_2S
	50	.49	2.1×10^{-6}	2.1×10^{-3}	Cu_2S
NaI	0.17	0.0011	9.0×10^{-4}	1.1×10^{-6}	Not Cu_2S
	2.0	.013	7.5×10^{-5}	1.3×10^{-5}	Cu_2S
	6.67	.045	2.3×10^{-5}	4.5×10^{-5}	Cu_2S
	15.0	.1	1×10^{-5}	1×10^{-4}	Very thin Cu_2S

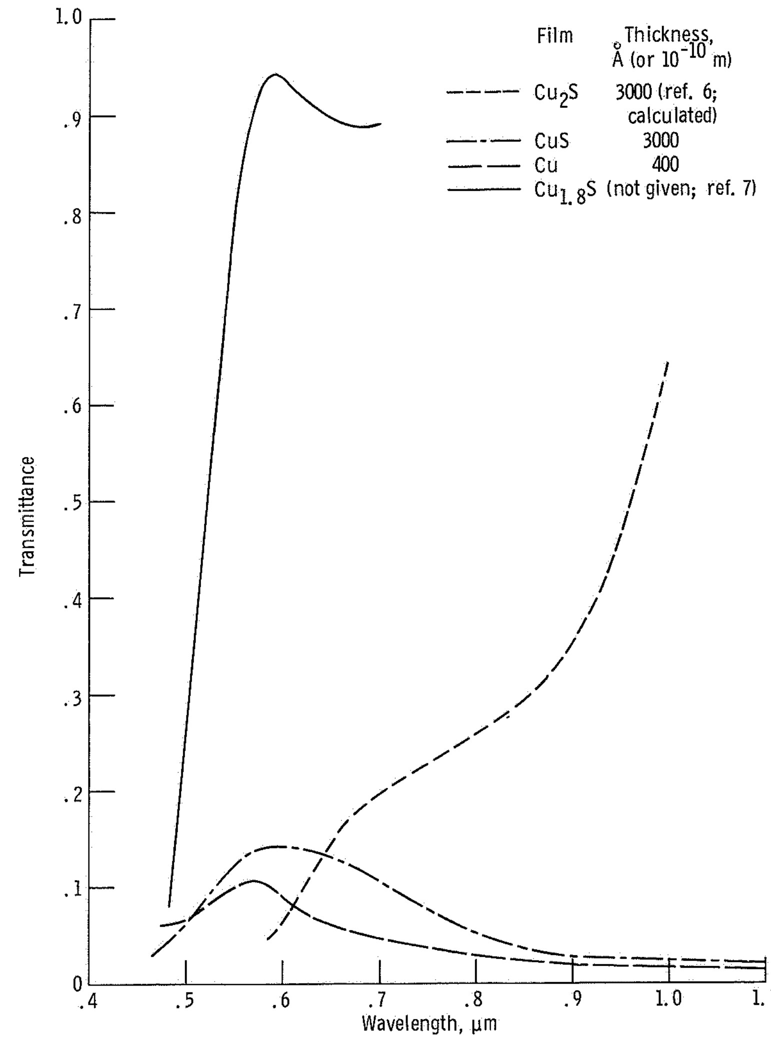

Figure 1. - Transmission of Cu_2S, CuS, Cu, and $Cu_{1.8}S$ films.

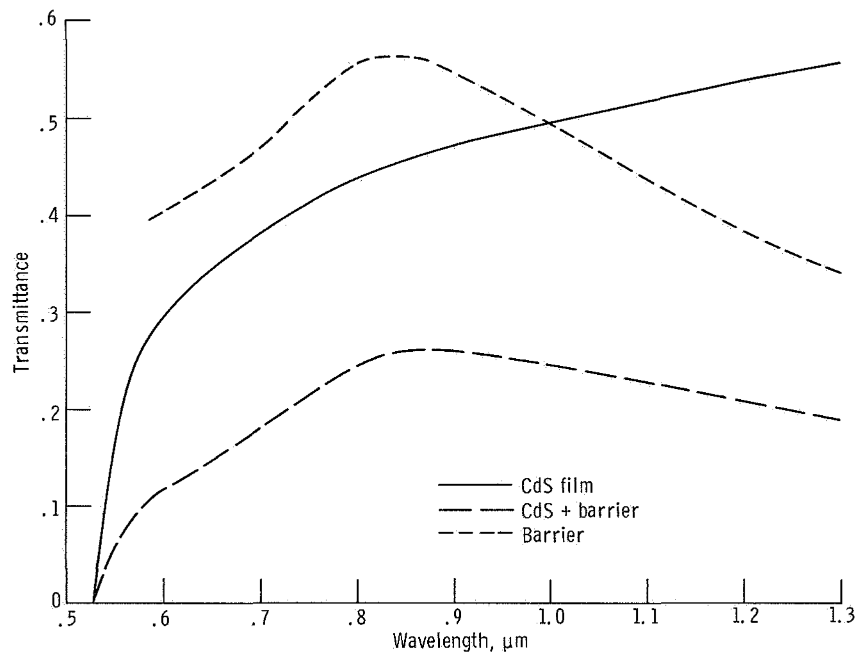

Figure 2. - Transmission of cadmium sulfide film plus barrier and of barrier.

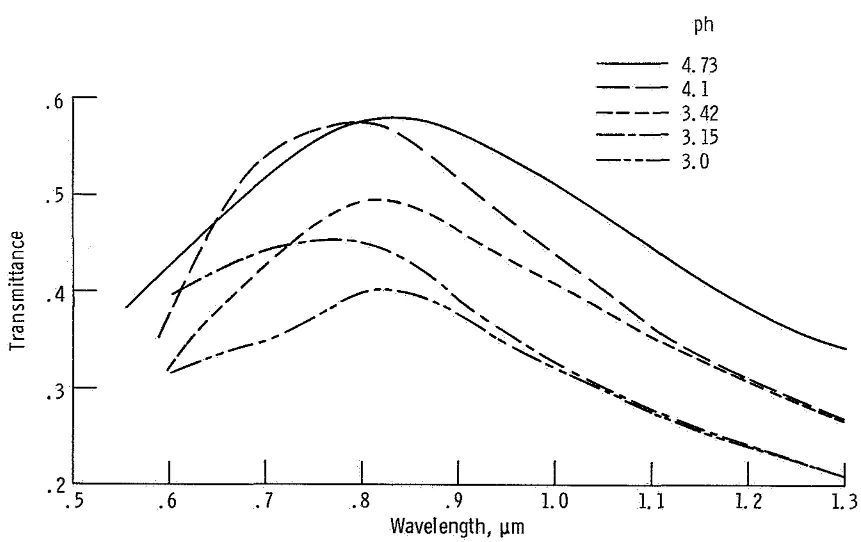

Figure 3. - Effect of pH on barrier transmission. Bath, 15 gram-per-liter cuprous chloride solution; bath temperature, 90° C; dip time, 5 seconds.

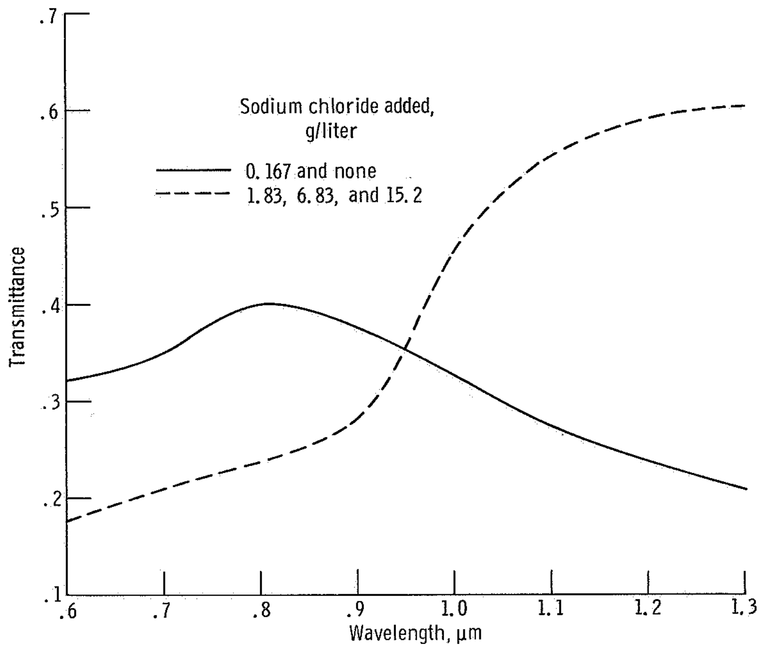

Figure 4. - Effect of sodium chloride on barrier transmission. Bath temperature, 90° C; pH = 3; dip time, 5 seconds.

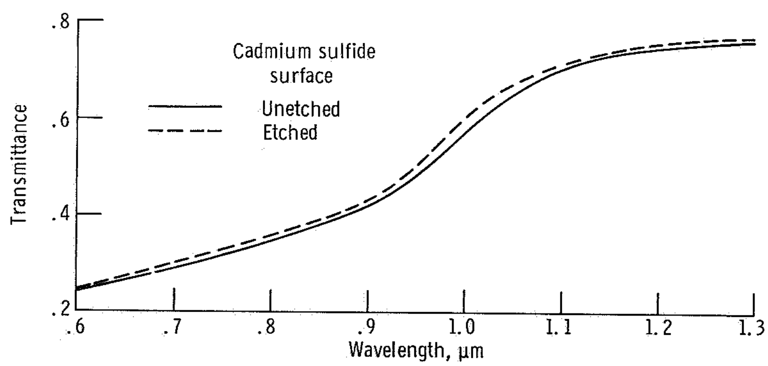

Figure 5. - Transmission of barrier as function of cadmium sulfide surface treatment. Etchant, 3 to 1 solution of hydrobromic acid and water; etched 5 seconds.

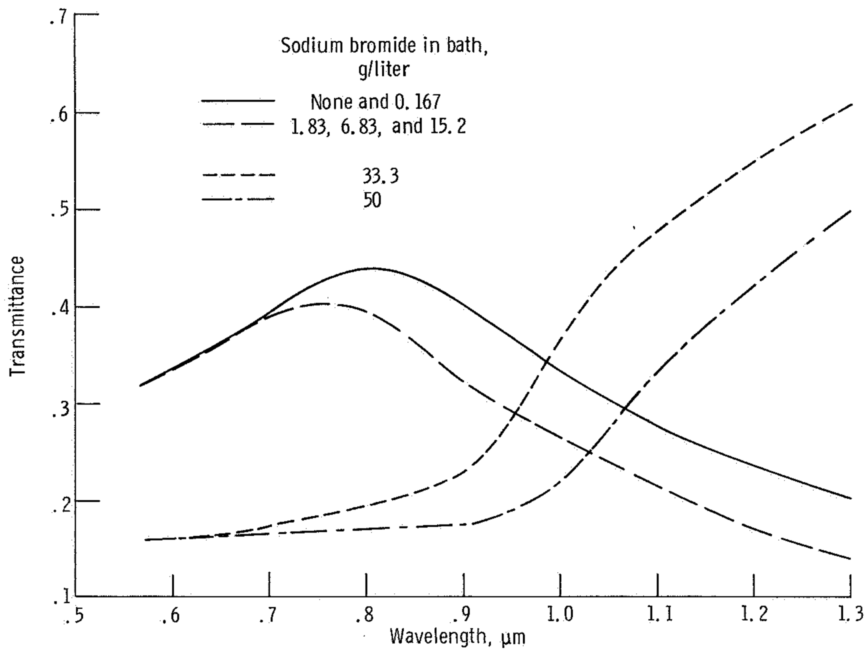

Figure 6. - Effect of sodium bromide in cuprous chloride bath on barrier transmission. Bath temperature, 90° C; pH = 3; dip time, 5 seconds.

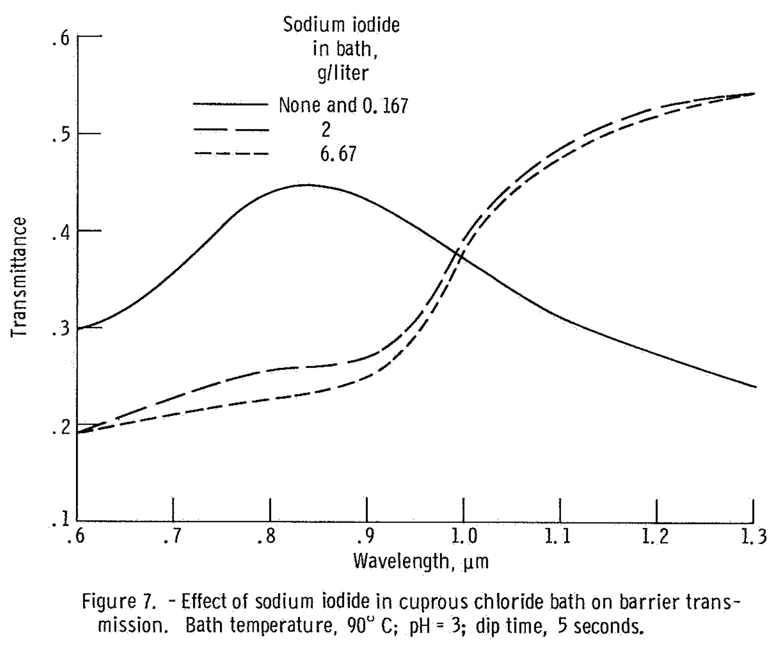

Figure 7. - Effect of sodium iodide in cuprous chloride bath on barrier transmission. Bath temperature, 90° C; pH = 3; dip time, 5 seconds.

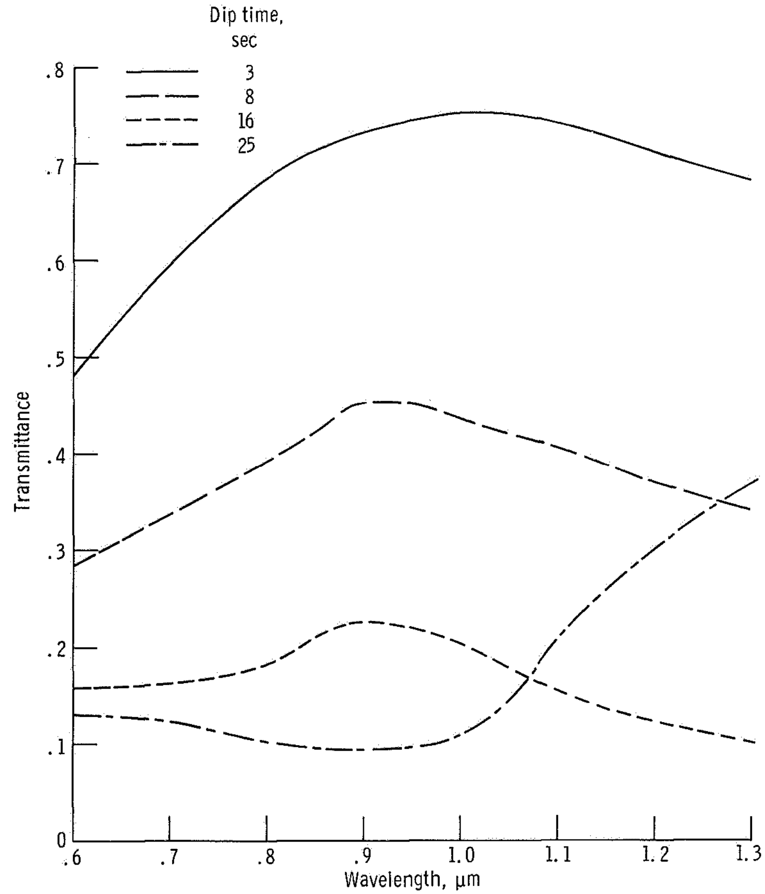

Figure 8. - Effect of dip time on barrier transmission. No sodium chloride in bath; pH = 3; bath temperature, 90° C.

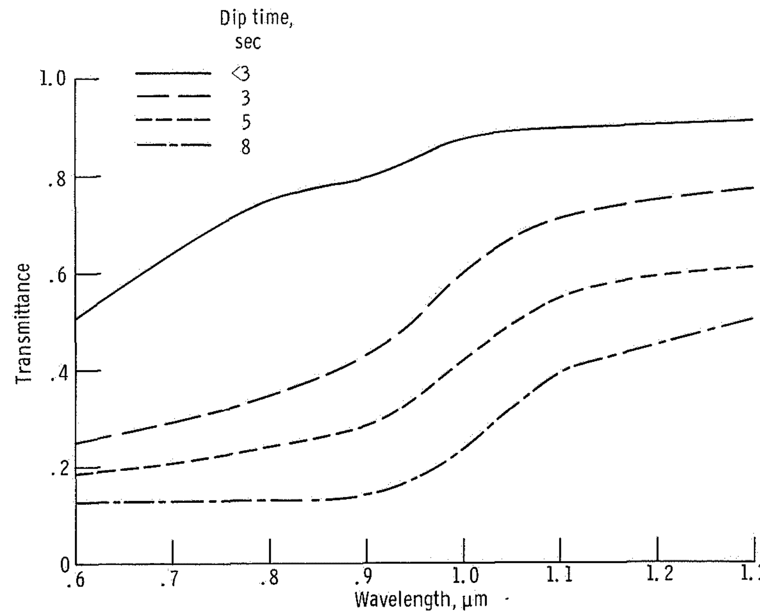

Figure 9. - Effect of dip time on barrier transmission. Bath temperature, 90° C; pH = 3; sodium chloride in bath, 15.2 grams per liter.

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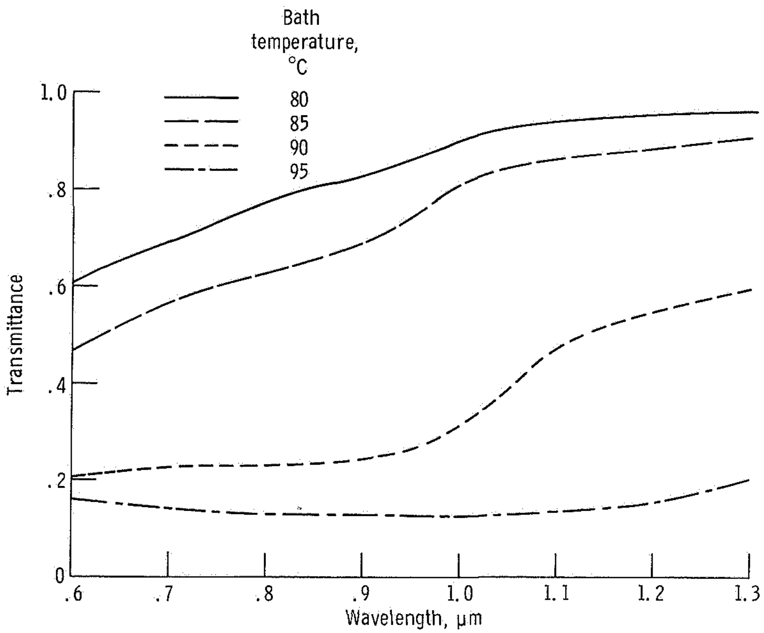
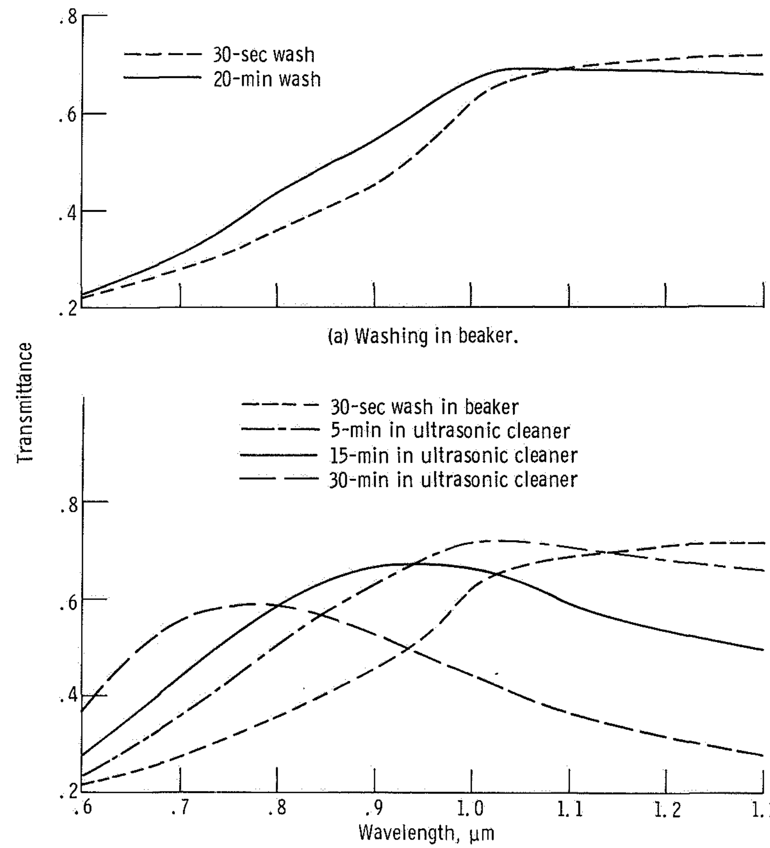


Figure 10. - Transmission of barrier as function of bath temperature. Dip time, 5 seconds; sodium chloride in bath, 15.2 grams per liter.



(a) Washing in beaker.

(b) Washing in ultrasonic cleaner.

Figure 11. - Transmission changes of barrier with washing in water. Temperature, 90° C; dip time, 4 seconds; 15 grams per liter NaCl in bath.

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