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CdSiAs₂ THIN FILMS FOR SOLAR CELL APPLICATIONS

Second Quarter Report: July 1–September 30, 1979

By
L. C. Burton
L. H. Slack

October 1979

Work Performed Under Contract No. AC04-79ET23007

**Virginia Polytechnic Institute and State University
Blacksburg, Virginia**

U.S. Department of Energy

Solar Energy

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ABSTRACT

Sputtered films have been formed with two improved composite (Si + CdAs₂) targets. Near stoichiometric and mechanically continuous CdSiAs₂ films have been formed on metal substrates. Films of this type will be used over the next reporting period for heterojunction formation.

Evaporation studies related to CdAs₂ have been completed. Stoichiometric CdAs₂ films could not be formed by means of single-source evaporation of CdAs₂ bulk. Hence, this dual source approach (Si + CdAs₂) does not appear feasible with the techniques used thus far.

Electron beam microprobe elemental analysis has been improved by changing from a GaAs standard to one of SiAs. The latter standard is more directly applicable to the compounds being measured. Accuracy of elemental composition measurements by means of the microprobe are estimated to be about ± 0.1 atomic percent.

A CdS/CdSiAs₂ junction was formed on bulk CdSiAs₂. This junction gives a good diode curve (no shunting or series resistance problems), although very poor photovoltaic response. Heterojunction formation and gridding procedure have been established.

The new Perkin-Elma Randex multi-target sputtering unit has arrived and is being installed.

Priorities for the next reporting period include fabrication of improved films and bulk discs for heterojunctions, more extensive fabrication and measurement of the heterojunctions, and installation and initial runs of the Randex sputtering unit.

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1. INTRODUCTION

This is the second Quarter Report for DOE Contract DE AC04 79ET23007, "CdSiAs₂ Thin Films for Solar Cell Applications." In this section, a brief review of the program is presented. Program tasks are then summarized, with emphasis on those treated during this reporting period.

1.1 BACKGROUND

The major reasons for studying CdSiAs₂ as a potential photovoltaic material were discussed in the First Quarter Report.⁽¹⁾ During that period near stoichiometric bulk polycrystalline CdSiAs₂ was synthesized by two techniques: (1) direct fusion of the elements and (2) direct fusion of the binaries SiAs, Cd₃As₂ and CdAs₂. The latter technique resulted in denser ternary material with good homogeneity. The binaries were found to melt congruently and were also formed by direct fusion.

Sputtered ternary films were formed using a bulk CdSiAs₂ target, and a composite target of CdAs₂ discs in a Si plate. Composition of the CdSiAs₂ target was found to change with sputtering time. Amorphous films deposited from that target were heat treated, and became crystalline and near stoichiometric but with poor mechanical properties. It appeared that films deposited from the composite target (Si + CdAs₂) could be adjusted to stoichiometry by means of sputtering power and target geometry. As deposited, these films also were amorphous.

With respect to evaporated films, a study of the thermal decomposition of CdSiAs₂ in vacuum was completed. The decomposition was found to be preferential toward Cd between 570° and 710°C, and toward As in the 710-1010°C range. It was concluded that evaporation of the ternary is not a suitable method for forming CdSiAs₂ films.

No heterojunctions were fabricated during the initial three month period of the program.

1.2 PROGRAM OBJECTIVES

The major objective of the program is the formation of CdSiAs_2 thin films suitable for heterojunction solar cells. The research effort is broken down into the following tasks⁽⁴⁾:

Task 1: Thin Film Growth

Task 1.1 - Preparation of bulk material for sputtering and evaporation. This includes CdSiAs_2 , CdAs_2 , Cd_3As_2 , SiAs and SiAs_2 as required to form targets and charges.

Task 1.2 - Sputter deposition of CdSiAs_2 films. Single and multi-target techniques will be used.

Task 1.3 - Evaporation of CdSiAs_2 films. Single and multi-source techniques will be used.

Task 1.4 - Film optimization.

Task 2: Film Characterization

Task 2.1 - Mechanical and electrical film characterization, including morphology, x-ray diffraction, average composition, composition profiles, resistivity and mobility measurements.

Task 2.2 - Wavelength dependence of absorption coefficient between 0.4 and 1.0 μm .

Task 2.3 - Minority carrier diffusion length measurement.

Task 2.4 - Determine film thickness for absorption of 50% and 90% of incident light as function of wavelength; optimize film thickness.

Task 3: Solar Cell Fabrication

Task 3.1 - Fabrication of $\text{CdSiAs}_2/\text{CdS}$ thin film heterojunction solar cells, using CdS as the collector material.

Task 3.2 - Evaluation of $\text{CdSiAs}_2/\text{CdS}$ solar cells by taking I-V curves

under solar or simulated irradiation.

Most of the research effort during the second three month period of the contract was directed toward Tasks 1.2, 1.3 and 2.1, i.e. toward thin film growth and characterization. These items are discussed in more detail in the following section.

2. TECHNICAL NARRATIVE

The research conducted over this reporting period was mostly associated with the bulk synthesis of CdSiAs_2 by solid state reaction and the establishment of a new standard for the electron beam microprobe; the RF sputter deposition of Cd-Si-As films from multi-component targets; the thermal evaporation of CdAs_2 and initial CdS/CdSiAs_2 heterojunction fabrication on bulk CdSiAs_2 .

2.1 BULK SYNTHESIS AND ANALYSIS

2.1.1 PRESSED DISC SYNTHESIS

Several important advances toward fabrication of bulk polycrystalline CdSiAs_2 have been made since the last quarterly report. The need to develop a method of synthesis of the ternary other than by direct fusion of the elements has been explained, and a unique solution was proposed. In the previous period, attempts to make good material were hampered principally by the lack of furnaces having uniformity of temperature across the reaction ampoule.

This situation was rectified by the installation and profiling of the new tube furnaces mentioned in that report. We were immediately able to start producing good silicon monoarsenide with this equipment. This SiAs was excellent in appearance and composition, and was adopted as the reference standard for analysis of the ternary compound.

The availability of good SiAs was one of the problems associated with the formation of ternary material from the binaries SiAs, Cd_3As_2 , and CdAs_2 . The work this quarter has been directed toward optimization of the process previously discussed.

We found that this type of ternary synthesis could not be performed in a single process step. The reason is that the SiAs is mica-like, having

a two-dimensional layer structure, and is very difficult to grind to particle size demension. Therefore, the SiAs was ground as much as possible and combined with the other binaries in the proper ratios, sealed in a fused quartz ampoule, and pre-reacted at 800°C for 4 to 7 days.

The ampoules were cooled and broken open. The resulting material was somewhat porous and fragile, and exhibited a major phase of CdSiAs_2 with a very minor SiAs content when analyzed by x-ray powder diffraction techniques. No other crystalline arsenide material was found to be present.

This material was reground and cold-pressed in a steel mold. The resulting material is 12mm OD by 2-4mm thick. These were then re-sealed in quartz and re-reacted at just under the CdSiAs_2 solidus temperature (821°C) for four days. No phase other than CdSiAs_2 was detectable in these pellets by means of x-ray analysis. One pellet was polished and subsequently used for CdS/ CdSiAs_2 heterojunction formation. A more detailed analysis of this pellet via electron beam microprobe, prior to heterojunction formation, showed the presence of a minor amount of an intergranular CdAs_x phase. This sample was coated with CdS and indicated heterojunction diode behavior (section 2.4).

The major reason for needing improved bulk CdSiAs_2 at present is associated with the fabrication of a bulk baseline reference diode. One problem with the bulk CdSiAs_2 up to the present time has been its porosity. We are working to reduce this problem. A present series in progress consists of eleven 12mm dia. by 3mm thick discs cold pressed from pre-reacted material. We are also working on upgrading the methods for production of higher quality binary starting materials.

2.1.2 Elemental Composition Analysis

There have been in the past some discrepancies between the compositions determined by means of electron beam microprobe and x-ray diffraction. This problem has shown itself as a deviation in the Si/As ratios for compounds synthesized as CdSiAs_2 and SiAs. This problem has been resolved by cross comparison of x-ray diffraction, wet chemical analysis and atomic absorption analysis with the microprobe data.

In analyzing the SiAs, one of the best and most homogeneous materials we have yet synthesized, a large discrepancy in accuracy was noted from true composition, although good repeatability of results was noted. Looking at the reproduction of the energy dispersive spectrum output (KEVEX), it is seen that the arsenic L_α complex and the silicon K_α lines are not sufficiently discriminated by the dispersion method to prevent overlap. In comparison, for the cadmium arsenides, where the L_α lines of each are quite different in energy, no such problem in analysis seems to be operating. Without a deep understanding of the dispersive methods and of the numeration procedures, it is sufficient to say that there is a pretty serious discrepancy here for the SiAs analyses. Further presuming the same problem to exist in the ternary analysis as an interference of the Si and As lines, one may make some calculations to correct the raw analyses.

One may thus determine factors to correct the silicon and arsenic microprobe data for SiAs back to an equimolar composition. Applying these factors to the corresponding elemental data for CdSiAs_2 , the corrected analysis gives a very good fit to the expected 1:1:2 ratio and is probably as good as can be expected from the microprobe.

An independent chemical and atomic absorption analysis of silicon arsenide bulk material has verified the composition to be SiAs. As a result of this, high quality cleaved SiAs has been substituted for silicon and gallium

arsenide as the primary standards for microprobe analysis. The resulting analysis of known ternary phase material gave good and (it is felt) reliable results. Many of the amorphous thin film compositions were reanalyzed or corrected on this basis.

2.2 Sputtered Thin Films

The procedures for forming the multicomponent sputtering targets were discussed in the last report, with initial thin film results. The work done to date using these targets is reported below, with all of the microprobe results obtained using an improved SiAs standard.

Four composite targets have been fabricated. The first was a silicon disc with a single CdAs₂ disc bonded at its center. It was used to characterize the geometry of sputtering from a single insert as a test of possible imaging effects between the two-phase target and the substrate plane.

The other three composite targets had Si/CdAs₂ area ratios equal to 1, 1.47 and 2.27. In the latter two, a 1mm thick 3" diameter silicon wafer was bonded between the Si/CdAs₂ composite layer and the copper backing plate. It was used to insure that the plasma never sputtered copper (or epoxy) from between the CdAs₂ inserts and the surrounding Si. The presence of this Si wafer was not found to have an effect on the sputtering process.

Sputtering from the first target (single CdAs₂ disc in Si plate) established that sputtering was a multicollision process between the target and substrates so that the Cd and As atoms were evenly distributed over the entire substrate holder area. This is indicated in Figure 1, where elemental compositions (determined by microprobe) are shown versus substrate location.

This experiment verifies that there is only a very slight imaging effect (as seen in the Cd distribution) and that several of these distributions superimposed would be expected to yield uniform composition across a distance of roughly 6 cm.

Films sputtered from the initial composite target (CdAs₂ discs in Si plate

Figure 1. Elemental composition of Cd-Si-As film sputtered from "bullseye" target, measured outward radially from center of substrate stage.

Table 1. Film Compositions for Films Sputtered
at 100 watts.

<u>Composite Target</u>	<u>Si/CdAs₂ Area Ratio</u>	<u>Composition (%)</u>		
		<u>Cd</u>	<u>Si</u>	<u>As</u>
#1	1.0	25.4	14.8	59.9
#2	2.27	12.7	28.7	54.1
#2	2.27	24.1*	23.3*	52.5*

* Heat treated at 600°C in Cd,As₄ ambient.

with Si/CdAs₂ area ratio of unity) were found to be Si deficient, as indicated in Table 1. This led to the construction of two additional composite targets with area ratios of 1.47 and 2.27. Data are also shown in Table 1 relating to films from composite target #2. It is seen that nearly stoichiometric films can result from target #2, subsequent to a post deposition heat treatment. These films are improved over those reported previously, in that they had far fewer mechanical defects after heat treatment. These films should be suitable for initial photovoltaic studies, which will be undertaken during the next reporting period (discussed in section 3).

The dependence of composition of films sputtered from the three composite targets is shown in Figures 2 and 3, for sputtering powers of 100 and 150 watts respectively. In both cases, Si content increases and Cd and As decrease as area increases. The decrease in Cd with increasing power is attributed to an increase in substrate temperature, as discussed in the previous report. The dependence of film composition on sputtering power (for composite target #1) is shown in Figure 4.

Composition of films has also been found to be dependent on substrate location. The compositional variation with position is most severe for Cd. This variation is shown in Figure 5 as a function of sputtering power and substrate position. The Cd content decreases near-center, at higher powers, apparently due to increased substrate temperature. Variation in Si and As compositions for the same film is indicated in Figures 6 and 7. In order to check on the possibility that the composition variation is caused (at least in part) by substrate temperature variation, temperatures were measured radially outward during runs using a thin film resistance thermometer. During a 150 watt sputtering run, the temperature was found to be 440°C at the substrate stage center, and 350°C at a distance 4 cm from the center. The decreased Cd content near-center (Figure 5) seems to follow this profile.

Figure 2. Dependence of film composition on Si to CdAs₂ area ratio of target. Sputtering power was 100 watts.

Figure 3. Film composition versus Si/CdAs₂ area ratio at a sputtering power of 150 watts.

Figure 4. Film composition versus sputtering power for composite target #1.

Figure 5. Dependence of film Cd content on sputtering power and substrate location for composite target #2.

Figure 6. Si content versus sputtering power and location for composite target #2.

Figure 7. As content versus sputtering power and location for composite target #2.

Cd-Si-As films have also been sputter deposited onto molybdenum substrates from composite target #3. The Mo substrates were of dimension 1cm x 1cm. In initial runs (substrates were only loosely attached to the substrate stage), the films were significantly low in Cd, even with respect to the glass substrate. This was apparently due to poor thermal contact with the water cooled substrate stage. Several 301 stainless steel substrates were deposited with Cd-Si-As films, and the Cd content was found to increase to 25% when a more intimate contact with the substrate stage was maintained.

2.3 Evaporated Thin Films

An interest in the deposition of cadmium diarsenide films has arisen as a result of the prospects of its use in co-evaporation of silicon to form CdSiAs_2 . The synthesis of the ternary film requires a careful characterization of the evaporation kinetics and stoichiometry of the binary end member. While a number of studies have been performed with the companion arsenide Cd_3As_2 as regarding thin film formation and characterization⁽²⁻³⁾ there has as yet been no appearance in the literature of corresponding data for CdAs_2 . To this end, the present study was undertaken to obtain order-of-magnitude estimates of evaporation and deposition rates.

Two phenomena which might be expected to affect the use of CdAs_2 in CdSiAs_2 fabrication would be: (a) non-uniform evaporation of the elements from the source due to differential vapor pressure effects; and (b) time-variant evaporation of the source material through surface-controlled rate effects. In the first case, a perturbation of the Cd/As ratio might result, while the second mechanism would lead to difficulty in establishing a consistent arsenide/Si ratio during co-evaporation. Furthermore, one would

expect the time required to volatilize a finite amount of evaporant would be strongly dependent on temperature, as would be the differential vapor pressure effect. Therefore one must seek temperature conditions which will permit evaporation chemistry giving a degree of control, yet a reasonable rapidity of completion, while concurrently optimizing the desired stoichiometry.

The purpose of this study was to see if all three conditions could be achieved using the CdAs_2 as the evaporant.

Modelling

In the analysis of the results it will be important to know what types of behavior to expect. The important quantities are:

- (1) film thickness buildup as a function of time;
- (2) evaporation rate as a function of temperature; and
- (3) film composition as a function of evaporation temperature.

It will help to develop some simple models to account for the likely progress of the evaporations. For case (1) above, presuming the evaporation flux to be constant and the evaporant source to be compositionally stable, the evaporant weight loss at any temperature (and hence the film thickness increase) should be able to be represented by an equation of the form:

$$\frac{dW}{dt} = a_1 t^2 + bt + c$$

where W is the evaporant weight; t is time; and a, b, and c are arbitrary constants. For case (2) above, the rate of evaporation (hence deposition) should be directly proportional to the vapor pressure of the source material. The most simple and usual description of vapor pressures above simple condensed phases has the mathematical form:

$$\log_{10} P = A + \frac{B}{T}$$

where P is the vapor pressure, T is the absolute temperature; and A and B

are constants.

Compared to this, modeling for case (3) above must be more sophisticated to account for real compositional variations, and hence will be discussed only generally.

Experimental

The cadmium diarsenide used was prepared by direct fusion from the elements in a fashion previously described (reference 1, pp. 23-26). The material was used in either the ingot form or crushed into a powder, and was evaporated using the apparatus set up for the ternary evaporation task⁽¹⁾. The evaporations were carried out from one gram samples at temperatures up to 625°C. Deposition rates were determined using a Kronos QM-300 series thickness monitor, and source temperature was recorded throughout the run. The substrates were not heated for the purpose of this experiment. A partial listing of the runs is included in Table 2. The following data were measured: film thickness versus time, deposition rate versus source temperature and film composition versus source temperature. The latter relationship is the most important, and is shown in Figure 8.

X-ray diffraction data confirmed the homogeneity of the starting material as cadmium diarsenide CdAs_2 , but also detected the presence of a Cd_3As_2 second phase in residues of incompletely evaporated source material. A few runs were made with heated substrates, but heating caused the coatings to be non-adherent or otherwise of poor quality. X-ray diffraction of the coatings deposited at ambient temperature failed to reveal any crystallinity.

Discussion

The amount of material used as evaporant, nominally 1 gram, was sufficient to produce a coating of 1 to 2 μm on the glass substrates. Since it was felt that for co-evaporation purposes this was a useful thickness range, all

Figure 8. Cd-As film composition as a function of evaporation source temperature.

the experimentation centered around this quantity of evaporant. Table 1 shows that at 625°C, the evaporation rate is slightly greater than a quarter-gram per minute. That is the amount of time to totally evaporate 1 gram would be less than four minutes at peak temperature. This temperature would most probably be too high for dual-source co-evaporation control with the possible exception of a sophisticated automatic system.

Table 2. CdAs₂ Evaporation Runs

CdAs ₂ Run	Temp °C	Type	Time	Wt. Loss (gm)	Average Rate of Evaporation
15	364	Slug	200 min	1.099	5.5×10^{-3} gm/min
14	Various	Slug			
13	555	Slug	7	1.19	1.7×10^{-1} gm/min
12	625	Slug	4	1.16	2.8×10^{-1} gm/min
11	476-80	Slug	27	1.28	4.75×10^{-2} gm/min
10	480	Powder	15	1.00	6.66×10^{-2}
9	480	Powder	5	0.725	1.45×10^{-1}
8	430	Powder	20	.826	4.13×10^{-2}
7	430	Powder	13	.563	4.33×10^{-2}

It is therefore clear that useful evaporation temperatures must be lower than this.

On the other hand, the evaporation carried out at 364°C required 3.3 hours to reach completion. Considering that the silicon would probably be being evaporated by electron beam gun, this would be quite too long to operate the gun for cooling. Hence the further evaporation schedule was held between these temperature boundaries, with a temperature of about 450-480°C seeming most appropriate timewise, according to Table 2.

Also indicated in Table 2 is the expected difference in evaporation rate from evaporation from a solid slug as compared to powder. At 480°C, the rate for the powder is about 60% greater than from a solid slug.

Furthermore, effusion from the evaporation source was noted at source temperature as low as 135°C. Therefore, it would seem unwise to try to use CdAs_2 as a heated substrate for deposition of other films above that temperature inasmuch as it seems to be susceptible to decomposition in that regime.

The time dependence of film thickness seemed parabolic or cubic in form, more demonstrably so at ~480°C than at ~364°C. The problem here in co-evaporation would be to match the deposition of arsenide with that of silicon to always insure stoichiometry, although an excess of arsenic might be advantageous, as is the case for GaAs evaporated films.⁽⁵⁾ Another possibility would be to deposit material somewhere near the correct atomic ratios, then post-process to improve crystallinity and stoichiometry.

The deposition rate did seem to nearby follow a logarithmic temperature dependence, as discussed earlier in terms of source vapor pressure. This data indicated the expected very strong rate dependence on temperature, as well as the possibility that more than one mechanism is present. The single most important result is that noted in Figure 8, which shows that in no circumstances was stoichiometric CdAs_2 obtainable from evaporation of CdAs_2 ingot or powder source material. Particularly, in the time/temperature region of interest, the discrepancy is about 3.5 percent of cadmium deficiency. Furthermore, these films could be graded in composition. Recalling that the x-ray results showed decomposition of the CdAs_2 to Cd_3As_2 during the runs, presumably arsenic is liberated first, to be followed by the cadmium. If this is so, the coating is nearly certain to have a graded composition. This again would make it very difficult to obtain stoichiometric CdSiAs_2 .

films by co-evaporation from dual sources.

One possible gross remedy for the non-stoichiometry problem would be to use a physical mixture of CdAs_2 and Cd_3As_2 powders as evaporation sources, with enough Cd_3As_2 admixture to enrich the cadmium content of the film to achieve an apparent overall Cd/As ratio of 1:2. However, this would probably not satisfy the chemistry requirements on a microscale without post-evaporation treatment. In any case, x-ray data indicate the necessity of post deposition heat treatment to develop crystallinity.

Conclusions

The evaporation of CdAs_2 seems to follow a polynomial time dependence and a semilogarithmic temperature dependence. Coatings were deficient in cadmium by $\geq 2\%$ so that no stoichiometric coatings were obtained. During the evaporation, the cadmium diarsenide partially decomposed to Cd_2As_3 α'' phase and a third phase, as well as probably free arsenic.

From the general results, it is concluded that direct formation of crystalline CdSiAs_2 thin films by co-evaporation of Cd/As material and silicon from separate sources, would be very difficult with equipment at hand.

However, it is also felt that there is still some hope for obtaining non-stoichiometric amorphous coatings of near-stoichiometric constitution, and developing the required quality by post-deposition thermochemical treatment.

2.4 Heterojunction Fabrication

Even though the objective of the program is a thin film device, an effort has recently been initiated to fabricate junctions on bulk CdSiAs_2 . There are two major reasons for this effort. One is to establish the technology which will subsequently be used on thin film cells. This includes the deposition of CdS, and contact gridding of the top surface. The second reason is to minor the photovoltaic properties of the bulk material and eventually, when the bulk material and interface are sufficiently optimized, form a base line with which to compare thin film cells.

We have recently formed a CdS/CdSiAs_2 junction on a pressed disc of CdSiAs_2 . This device was formed as follows: the bulk CdSiAs_2 formed as described previously⁽¹⁾; a disc was cut, polished and mounted on a copper backing plate with silver epoxy; CdS was deposited on the CdSiAs_2 surface to a thickness of about 5 μm , at a substrate temperature of 200°C; an indium grid was evaporated through a mask onto the CdS. This structure is shown in Figure 9.

It should be noted that due to the mechanics of the bulk CdSiAs_2 formation, the material is somewhat porous, and the resulting disc surface contains some fissures and other depressions.

Several results were obtained from measurements on this device. These are discussed below. (This device was actually fabricated in early October, and only initial electrical measurements have been made. These measurements will be expanded, and additional devices made, in the next period, and reported upon.)

A good diode characteristic was obtained, indicating rectification between the CdS and CdSiAs_2 and ohmicity of the contacts. We are in the process of measuring the current-voltage relation in more detail to gain

Figure 9. Cross section of CdS/CdSiAs₂ heterojunction made on bulk CdSiAs₂.

information about the nature of the interface. No shorting or series resistance problems are apparent.

The junction exhibits a very poor photovoltaic response. The device has not yet received post-fabrication heat treatments of any kind. Attempts to improve the response by techniques such as this will be delayed until a larger number of devices are fabricated on the newer type of discs (discussed in section 2.1) during the next period.

Equipment Improvements

The new multi-target sputtering system (Perkin-Elmer Randex 2400 8-J) has arrived and is being installed in a dedicated room. After installation this system will be used to deposit CdSiAs_2 films, in order to help achieve the objectives of this contract. Progress of work related to this machine will be reported in the monthly and quarterly reports. The installation is expected to be completed by mid-November, including a two-day on-site visit by a Perkin-Elmer engineer.

3. NEXT QUARTER PLANS

The major effort will concentrate on obtaining crystalline sputtered films for heterojunction fabrication. Films will be sputtered from recent composite targets onto metal substrates. Improved substrate mounting procedures are anticipated to result in more uniform films with better stoichiometry. Post-fabrication heat treatments will continue to be used to improve the films.

CdS/CdSiAs₂ junctions will be formed on both recently made bulk discs, and on films deposited onto metal substrates. Procedures outlined in section 2.4 will be used for heterojunction fabrication. More extensive measurements will be made on the devices.

The new Perkin-Elmer Randex sputtering unit is being installed in Holden Hall. It is anticipated that multi-target sputtering runs will be performed and reported during the next reporting period.

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