THIRD SEMI-ANNUAL PROGRESS REPORT

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X-RAY TRANSMISSION MICROSCOPE DEVELOPMENT

Period of Performance
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1. Introduction

This report covers the third 6 month period from February 28, 1995 to August 31, 1995 under this contract.

The main efforts during this period were the construction of the X-ray furnace, evaluation and selection of the CCD technology for the X-ray camera, solidification experiments with Al alloys and Al-zirconia composites in the prototype furnace, evaluation of specimens for the particle pushing flight experiment - PEPSI, measurements of emitted spectra from X-ray source, testing of the high resolution X-ray test targets, and the establishment of criteria for and selection of peripheral equipment.

In addition to these tasks, two presentations were prepared in this period; one for the AIAA Microgravity Symposium and another for the Gordon Conference on Gravitational Effects in Phisico-Chemical Systems.

2. Project Objectives

The objectives of the NASA ATD consist in the development of an X-Ray Transmission Microscope (XTM) for the in-situ and real-time observation of interfacial processes in metallic systems during freezing or solid-solid transformation.

The performance goals established for the XTM are:

The XTM for solidification studies should have the following capabilities:

1. provide a resolution for specimen features of 10-100 μm,
2. at solidification rates of 0.1 to 20 μm/sec,
3. temperatures up to 1100 C with temperature gradients up to 50 C/cm,
4. with contrast sensitivities sufficient to detect 2-5% difference in absorptance,
5. offer 1, 2 and 4 in exposure times of a few seconds, and
6. permit recording of stereo pairs for depth information.

To date, many of these goals have been reached:

Cellular structures have recently been observed during solidification of Al-Ag alloys with dimensions of 50 micrometers width or less, thus point 1 is satisfied. Growth rates of 0.2 μm/sec at the low end are presently possible with velocities of up to 34 μm/sec having been reached. This range of translation rates is possible only by employing two different motors. The new furnace drive should provide the wide range of translation rates specified with just one motor in point 2. The temperature gradient in the prototype furnace is about 45 deg/cm while the hot zone temperature limit is presently 890 C in air atmosphere. The parameters of point 3 will be reached in the new furnace but an inert gas (nitrogen or argon) atmosphere will be required. Using the 12 bit cooled CCD camera from Photometrics and the Thompson CSF X-ray image intensifier for the converter, a 3.5% contrast level sensitivity was achieved using the high definition X-ray source and detection system with large features. Although contrast is feature-size dependent and impacts resolution, significant improvement in this value is anticipated with the new technologies of converter and camera to be purchased.

The circumstances leading up to the camera acquisition are outlined in the following sections.

3. Optimization of X-Ray Converter/Camera for Solidification Studies

3.1 Introduction

A critical component of the development of the solidification XTM is the detection of the X-rays and conversion to a useful image. Four technologies, all based on CCD's, were evaluated for the X-ray camera/converter. Although some aspects of these technologies are several years old, the selection of one as the best for this application could not be made based on specifications or claimed performance. The oldest technology was the X-ray image

intensifier for image conversion. On coupling this device with the cooled CCD camera, the best images possible from this technology were obtained. The other three technologies are based on intimate coupling of the converter and CCD itself. In fact, one promising method uses the CCD silicon itself as the X-ray to electron converter or detector. The remaining two approaches put a scintillator in front of the CCD and converts the X-ray photons to visible light photons. These in turn are converted to electrons in the CCD and stored in the pixel elements for later read-out. Comparisons between these technologies have shown that contrast, resolution, signal to noise ratio, and sensitivity must simultaneously be examined. Figure 1 shows the three CCD technologies.

The first of the three technologies, the direct CCD converter only became available a couple of years ago. Since then, it was shown to be a contender to the intensifier method. This technology uses the CCD as the X-ray photon converter and as the storage device for the converted image so it can be displayed. The second and third CCD technology would be to use a cooled CCD for image collection from the visible light output from a scintillation screen which converts the X-rays to visible light. The detection sensitivity with this technology is the difficulty. Resolution and contrast are not as significant a problem. Either the scintillator, here a CsI phosphor, is placed directly on the CCD surface or on a fiberoptic face plate which is then bonded to the face of the CCD. One of each of CCD was obtained as engineering grade (or having a few known faults) from EEV in England. These were the smallest sized (0.85 x 1.2 cm) devices (model CCD 02-06) with 578 x 385 pixels of 22 μm size. Camera readout electronics were also purchased for the evaluation. Cooling the CCD was not a requirement for this evaluation so only 8-bit images were obtained.

3.2 Progress in Evaluation of X-Ray Intensifier Technology for X-Ray Conversion.

For a year and a half, testing and improvements in the XTM system have been made, particularly with intensifier imaging. The emphasis during the last 6 months was the evaluation of new conversion/camera technologies using engineering grade CCDs in a breadboard style camera. Much of the evaluation is based on comparison with the capabilities of the intensifier system.

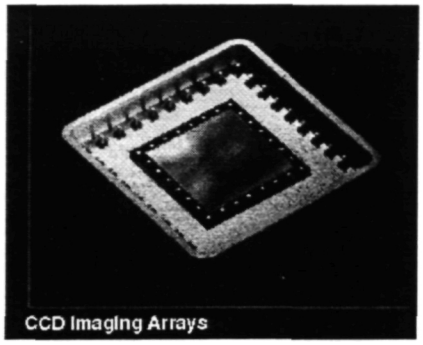
The caliber of our recent solidification experiments with aluminum alloys of various kinds has been limited (as anticipated in our proposal) by the X-ray intensifier technology. The limitation is most apparent when attempting to observe the finest microstructures. The best results are achieved consistently when the magnification and flux are as high as possible and the acceleration voltage of the X-ray source is above 50 kV. No useful images can be obtained below 30 keV because that is the lower energy cutoff of the intensifier. Our initial configuration for the intensifier tube is a tri-field type which permits higher magnification through electronic means within the intensifier. The penalty here is a less bright image. In other words, the intensifier gain is reduced proportionately, thus, only with high fluxes and stationary objects could these electronic magnification factors be of benefit. There is a marginal improvement in contrast and no improvement in resolution (since the cathode coating is the limiting step) when electronic magnification is employed. Our conclusion is that the tri-field capability is not optimum for real time solidification studies for microstructural features smaller than about 100 μm.

The magnification data presented in this report were made with the intensifier mounted at 550 mm from the X-ray source. Thus, with the cameras used, the magnification is known accurately based on distance from the source.

With alloys of high internal feature contrast (like Al-Pb alloys), much can be observed by using the uncooled video CCD camera. This detector allows true real-time observation of the a moving sample (instead of exposures of several seconds required for the higher resolution cooled detector system). Scanning through the specimen in real-time provides an extraordinary appreciation for the 3-D phase structure of the sample. A sense of depth is perceived (due to the parallax effect) during lateral scanning since the images of the different strata within the specimen shift at different speeds. The state-of-the-art optimized converter/camera to be acquired may allow both the highest resolution and real-time observation. However, the frequency of the image frame rate will depend on sensitivity, magnification and flux. Certain conditions like low flux levels, high magnifications, slow A/D conversion rates and low energies may not offer smooth movie-like image rates with the new camera. If it is not feasible to track large image displacements with the new camera, it will be necessary to provide for interchangeability of the two methods of image conversion.

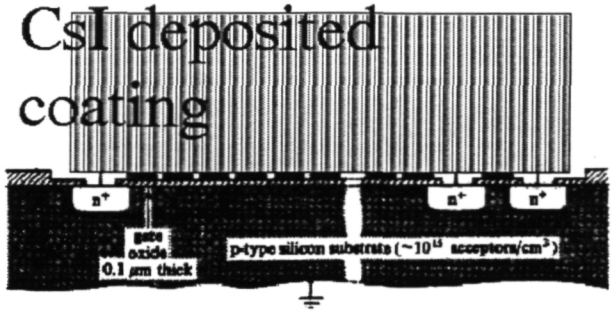
Presently, we are using the state of the art X-ray image intensifier from Thompson CSF which was purchased with the Fein Focus Inc. apparatus. A cooled CCD camera, having 1000x1300 pixels with 12 bits per pixel capability was coupled to this intensifier. This camera provides a much greater dynamic range and permits extended exposures to be made compared to the uncooled CCD camera normally supplied which is much like those used for closed circuit TV. The uncooled camera cannot always provide the image quality required for solidification studies but does offer movie-like video images. With higher contrast specimens like the Al-Ag alloys, interface morphology is readily viewable with the video camera. At best, such a camera only provides 256 gray levels as compared to the 4000 from the cooled CCD. A more natural sense of the interface kinetics is obtained with these higher image rates. The incentive for high frame rates is thus significant to any camera technology selected. The three detector technologies discussed next are independent of the intensifier method.

Figure 1. X-Ray CCD Technologies for XTM



Direct X-Ray Conversion in CCD

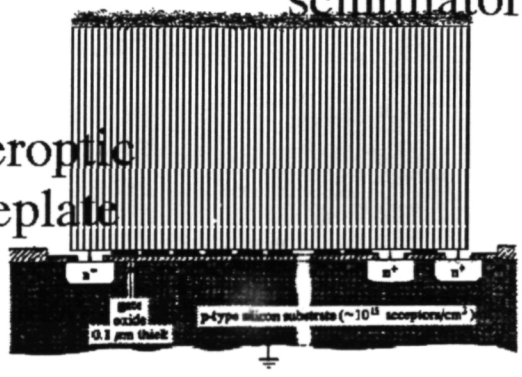
CsI deposited coating



CsI Coated CCD

scintillator

fiberoptic faceplate



CCD with FOFP and particulate scintillator coating

3.3 CCD Technologies Addressed

The first is a CsI coated CCD which offers as many as 16 bits per pixel dynamic range in the most elaborate configuration. The CsI coating converts the X-rays to visible light which is directly coupled into the CCD. The coating should shield the CCD from transmitted X-rays and protect the CCD from radiation damage while minimizing spurious signals caused by X-ray photon absorption in the CCD elements. However, thicker coatings increase X-ray absorption (DQE or efficiency) while decreasing image spatial resolution (MTF).

Another promising technology, similar to the above, is the addition of a fiberoptic faceplate to the CCD which can in turn can be coated with the scintillator material which may not be CsI but another, perhaps more efficient phosphor material we have been evaluating. This method allows the X-rays passing through the phosphor layer to be blocked by the glass faceplate before damaging the CCD. The phosphor coating is made as thin as required to provide adequate resolution and not required to stop all the X-rays from passing through to the CCD. Coupling losses are increased but spurious signals from stray X-rays getting into the CCD elements is nearly eliminated. It was decided to use the CsI coating on the faceplate since it can be deposited in a thick layer of 100-300 microns while still retaining high resolution since the crystallization (deposition) structure is needle-like. Particulate phosphors would only be useful in layers of less than 50 micrometers thickness since visible light scattering within the layer would quickly reduce resolution since the most excited upper surface of the phosphor layer would be positioned even further away from the CCD elements. The phosphors would have been somewhat superior than CsI were it not for this factor.

A third and recently developed detection method we were testing is the direct conversion CCD technology where the X-rays are detected by absorption in the silicon of the CCD itself. Silicon has a low cross-section compared to the scintillator materials and so will not stop as many X-rays providing less signal. To compensate for this, the silicon layer was thickened from the typical 5 micrometers to 50 micrometers to capture more X-rays. This method offers the best resolution since the pixels define the image elements and little scattering is incurred within the CCD (compared to thick phosphor coatings). Earlier versions of this technology were evaluated and shown to be viable. However, the efficiency of this technology favors the lower range of photon energies that are most beneficial for the light alloy solidification studies. X-ray hardened CCDs have only been practical in the last few years. Even for these CCDs some damage occurs, but the CCDs will have a significant life before the S/N ratio drops to unusable levels. While this latter style CCD is X-ray hardened, the CCD used for the former two technologies is not. Should X-rays pass through to the silicon layer, if they are absorbed, spurious X-ray induced signals would be created. This is actually an undesirable condition. As shown in the comparison figure 2 the direct conversion CCD technology offered a high resolution and contrast (MTF). The lower efficiency of detection and the low fluxes generated by the X-ray source at the low accelerations where this device works best (<20 keV) made this a second choice. Were the funds available, a second camera with this type of detector would be recommended.

3.4 Comparison Between Technologies

These competing technologies are not clearly delineated by performance for our innovative applications. A comparison using manufacturer's data is possible only between CsI coated and direct conversion CCDs. The result from this data is that the best S/N ratio available depends on the X-ray energies involved.

Operation of the X-ray source with low energies offers higher contrast regardless of the detector. We found the optimum energy for aluminum alloy systems is 35 to 100 keV acceleration voltage. The thin specimens do not attenuate very highly and so low energy use is an option. The dynamic range desired depends on the number of X-ray photons detected. The direct conversion approach may not permit as many photons to be detected to give the desired dynamic range. This comes about from the large number of electrons created per absorbed X-ray photon ($\sim$300). Full well electron capacity of the pixel is set by its volume and is limited to about 500,000 for the devices we are interested in. The dynamic range is simply the number of detectable photons per pixel or 500,000/300 = 1666. This offers at best a range of 10 to 11 bits per pixel. The other methods offer up to 16 bits where an estimated 5 electrons would be created from each absorbed X-ray in the scintillator layer. This in turn means that more X-rays are needed to fill the well or to obtain the dynamic range desired.

Resolution from any of these CCD methods will be far superior to the image intensifier approach (manufacturer specification of 3 lpmm). However detection efficiency may determine the optimum technology, since our requirement is real time detection of solidification microstructures.

Based on the evaluation, a final selection of the CCD type and specifications for the optimum detection system for solidification studies was made. The selected advanced technology CCD will have the largest number of pixels available (EEV CCD 05-30 with 1242 x 1152 pixels) and be of the highest grade. The advanced technology readout electronics will permit as many as 16 bits (128,000) gray scale resolution per pixel and a 1 Mhz clock rate at 14 bits per pixel. The selected CCD will have to be cooled to -50 C and sealed in an air tight housing to obtain the optimum performance level. Digitization will be performed in the computer and the images stored and subsequently enhanced. The advanced technology X-ray camera is expected to cost about $50,000 where $12,000 to $15,000 of this will be the cost of the selected CCD itself.

3.5 Results of the Evaluation of the Three Technologies

Each of the three CCD devices was placed in the camera and the line-pair gauge placed on top of the detector to provide a consistent input. Exposures were made with 50 kV and 350 μA at the source for 0.1 sec and the CCDs were 5 inches from the source. The sample images for each are shown in Figure 2. The resolution of the gauge is 22 lines per mm at the small end and was satisfactorily resolved in all three cases. In the figure, the last marker is the 20 lpmm level. Earlier experiments by Dr. Kaukler with particulate phosphor coatings delivered only a 16 lpmm resolution. How well the lines are resolved is the best indication of which method is best. Histogram plots for each image showed the relative sensitivity differences. The brightest image was obtained from the CsI coated CCD while the direct conversion was the least bright for equivalent exposures.

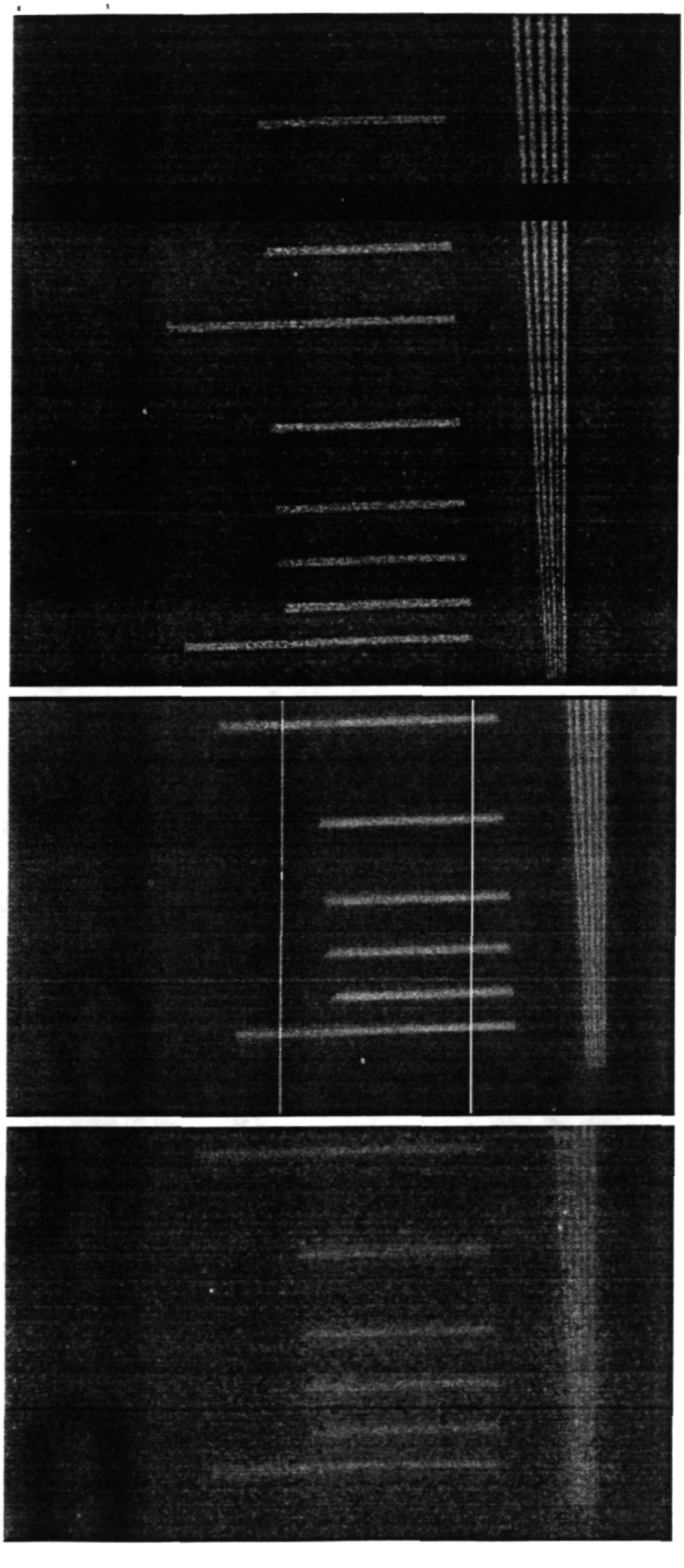


Fig. 2 Comparison of Image Resolution for Three X-Ray Detector CCD Technologies

Direct Conversion, 50 kV 350 μA

CsI Coated Fiber Optic, 50 kV 350 μA

CsI Coating on CCD, 50 kV 350 μA

4. Custom Manufacture of Suitable X-Ray Resolution Gauges

An accurate resolution X-ray test target for feature sizes 2 μm or less is essential for comparative tests of the candidate instrument configurations. Custom ion milled gold and nickel resolution targets were procured and evaluated. Three such gauges were delivered and each evaluated with various microscopies to verify quality and calibration. Each of the three differ slightly but all provide the limiting resolution data.

Such gauges are not offered by the X-ray source manufacturer, nor could they provide a source. A search of the few companies in this country that could perform the task resulted in these items for our exclusive needs. The targets are glass plates with about 3 micrometers of metallization into which a standard pattern (USAF test target) was etched by ion milling. It is technically difficult to produce fine features that preserve the feature shape throughout a thickness that is greater than the width of the feature and especially to do so with heavy metals like gold. (The technology is well developed with silicon for electronics.)

When successfully done, sufficient contrast is available to view the small, detailed pattern with X-rays. The pattern provides increasing line pair resolution to the limit of the X-ray source which is our tests show is better than 2 micrometer resolution of line pairs.. The gauge permits measure of the resolution and calibration of the instrument before use. These measurements have shown that adjustment and calibration of our microfocus X-ray source is frequently required (nearly daily) to maintain the ultra-high definition capability of this instrument.

5. Progress in Development of an Optimized X-Ray Transparent Furnace for Solidification Studies

5.1 Introduction

We have begun the assembly of an X-ray transparent furnace for real-time solidification studies that will be optimized to provide the highest image magnification feasible. Extensive testing utilizing the prototype furnace has yielded information that have enabled us to establish the specifications for the transparent (to) X-ray furnace to maximize potential science return. The prototype furnace enables a maximum real-time magnification of the solidifying sample of about 50 X. In order to obtain (the first) real-time images of the growth of fine microstructural features (as the fibers in eutectics and monotectics) we require a magnification of the order of 200 X. The primary difficulty in obtaining the higher magnification is the requirement to place the molten metal sample safely within a few millimeters from the X-ray source without overheating the X-ray source.

It was desired to have an operation temperature of the furnace such that metals like silicon or copper could be melted and solidified. This placed a constraint of having the furnace capable of operation in the hot zone of 1200 degrees C. It was also desired to not require vacuum operation. So provision was made for inert gas operation or even atmospheric.

Our scientific research with the XTM have shown that an excellent range of fundamental solidification phenomena unobservable by other methods can be studied using aluminum alloys. The added advantages of aluminum alloys are the low melting temperature

(660 C) and the commercial interest in such alloys. There is a direct trade off in furnace design between maximum magnification and melting temperature. From the fundamental solidification science perspective, the need for higher real-time magnification is quite evident. This drives the priority for furnace design to offer the highest magnification for these alloys so that the smallest solidification structures possible can be observed and measured. It turns out that higher magnification also offers a higher image quality at the converter/camera such that faint objects can be seen better with higher magnification even though the resolution requirements are less stringent.

To date, within the limits imposed by the prototype furnace, some of the solidification structures we set out to find have been seen. In particular, the cellular structures caused by planar interface instability due to constitutional undercooling have been clearly seen. The onset of the instability was also observed. Planar interfaces which are normally phenomenologically poor, sometimes show, under the XTM, interesting features like grain boundaries that intersect the solid-liquid interface. Figure 3 shows these features in a composite of three interface views. However, examination of the samples post solidification, when the magnification is not limited by the furnace structure, show even more structure and detail. This comes about from the higher magnifications available when placing the specimens close to the source. Some features, like the fibrous structures in the Al-Pb monotectic are so fine (about 5 micrometer diameter) that they could not be resolved during growth using the prototype furnace. To be able to view the formation of these structures and similarly small structures like dendrites we will require this new furnace design.

A secondary, and important benefit from being able to bring the specimen close to the X-ray source is the improved contrast or gray scale resolution. This comes about from the limited MTF or modulation transfer function the intensifier has. The resulting enlargement of the features reduces the net contrast losses making image details less diffuse. The inherently high MTF of the CCDs offer an advantage to this problem, but it will not go away completely regardless of what technology is used. Therefore, good furnace design is nearly as important to getting the best images as is the detector technology.

5.2 Furnace Design Criteria

To safely bring a 1200 degree heating element within a few millimeters (a quarter of an inch) of anything temperature sensitive is a difficult task. There is no insulation that adequately protects in such thin gaps. It was decided to use flowing, chilled water to protect the outside of the furnace. The penalty of this approach is that the heating element must now heat the water as well as the sample. Details of the materials selection and geometry were the most time consuming tasks. Both thermal radiation and conduction are the modes of heat transport at these temperatures. So some insulation was required. The new furnace can allow only one fifth the space for insulation compared to the prototype furnace. It was decided that the upper surface of the furnace be made one millimeter thick or less out of copper alloy foils sandwiching cooling water. The balance of the gap would be fibrous ceramic insulation capable of withstanding the element temperatures continuously.

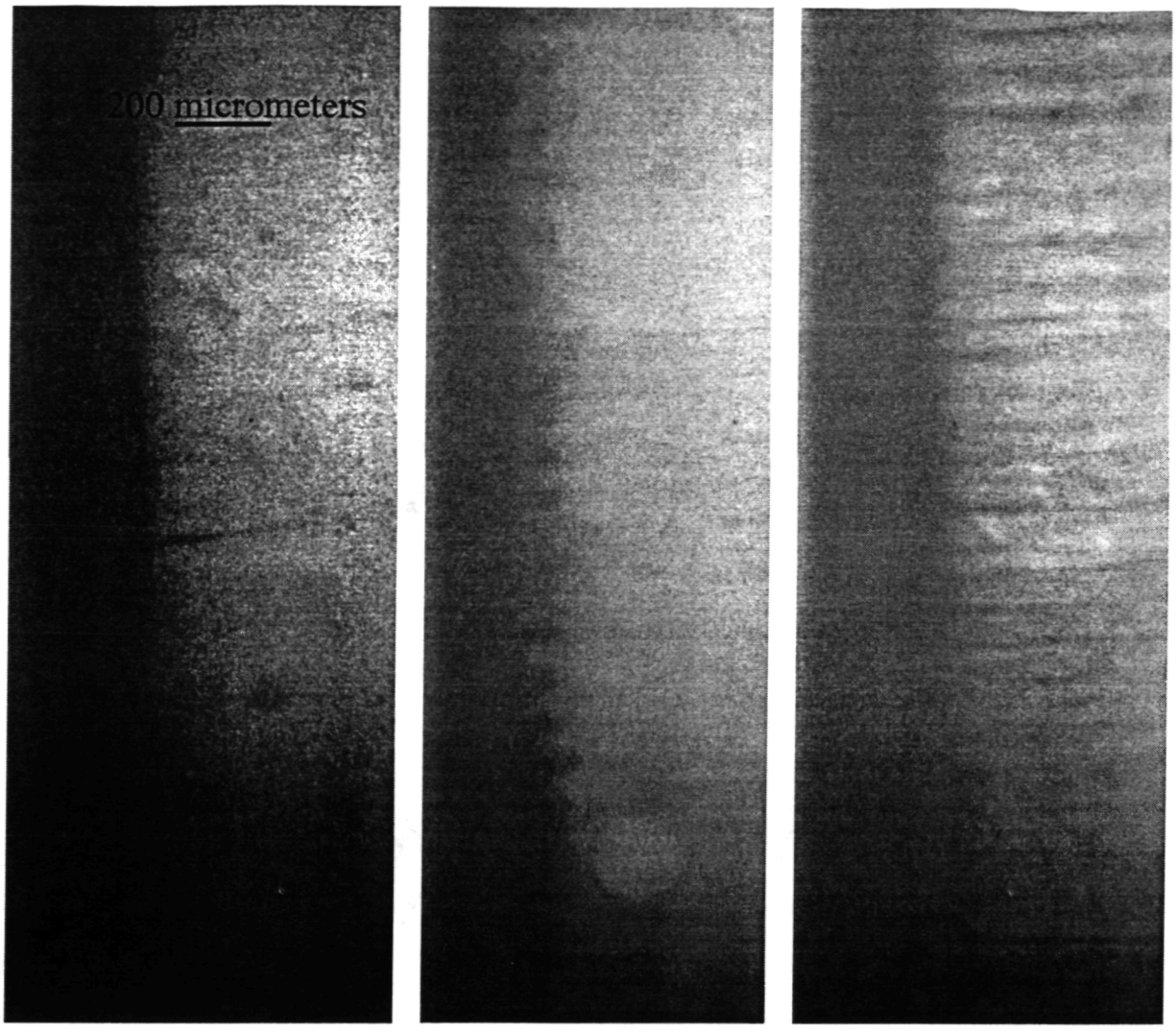


Figure 3. Al 2% Ag interfaces. Each are 2 second exposures at 55 kV acceleration and 200 μA current. Left figure shows planar interface growing at 1 μm/sec; grain boundaries intersect the interface isotherm. Middle figure captures the initial stages of cellular breakdown after the rate was increased to 2 μm/sec from 1.5 μm/sec; void was engulfed in lower segment. The right figure shows the steady state cellular growth at 2 μm/sec.

6. X-Ray Spectrum Measurement (Preliminary X-Ray Source Evaluation Continues)

One important aspect for modeling the specimen response to X-rays is the knowledge of the emission spectrum from the X-ray source. This information was not available from the manufacturer for our high definition tube. (It is reasonable to not expect such detailed information for such a device when performance for the intended nondestructive testing applications don't require it.) However, accurate utilization of the X-ray transmission models developed in the first year of this ATD depends on this data. The models prepared earlier in

this work used published spectra for more common X-ray sources using a tungsten target. It was reasonable to expect similar response from any tungsten target illuminated with high energy electrons but it turns out the spectra do differ significantly.

To obtain our X-ray source spectra, a multichannel analyzer and room temperature X-ray detectors of Cd-Zn from EEG was borrowed from the CCMR at UAH to perform the measurements. The test data revealed that the fluxes emanating from the tube even at minimal operating current were swamping the detector. With care, the X-ray source was detuned to produce a low enough flux yet still produce the characteristic spectrum. The useful energy range of the detector is between 14 keV and 200 keV. This was adequate for our work. A set of spectra were obtained without concern to flux level since the overall shape of the curve was all-important. Actual flux measurements would not have been possible given the sensitivity of the detector to the highly abated fluxes. It was not expected that the spectra would differ from the published curves to the degree they did. The overall difference was in the peak energy for the given acceleration energy. In all cases, the real peaks were shifted to lower energies than the general published data. In addition, the higher energy characteristic peaks often seen for tungsten were not found. The measured spectra for our micro-focus X-ray source are shown in Figure 4.

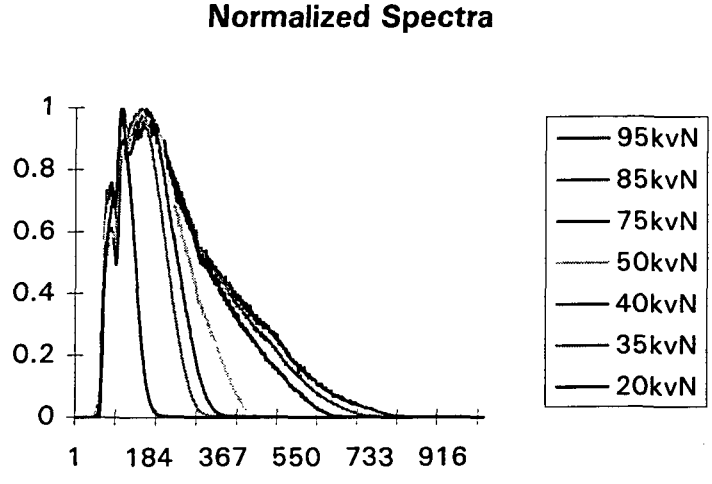


Figure 4. Normalized spectra from Fein Focus High Definition Tube

7. Mathematical Modeling and Solute Gradient Measurements

The X-ray absorption model developed in the first year needed to be examined more closely when it was observed that the calculated absorption level of silver was comparable to that of lead for equal thicknesses. To predict particle contrast, a 10 micrometer layer of the particle material on 1 mm Al is assumed and compared to 1 mm Al. The calculations **unexpectedly** showed similar absorption from these two elements. Closer examination

showed that the molar density of the material plays a role in the absorption calculation in a way that alters the weighting of the atomic absorptions. The unexpected outcome of this analysis is that the selection process for the alloying elements is more complex than first thought.

The models cannot account for absorption when the elements are in solution unless the molar density is known. As a result, solutal measurements will be needed that support future prediction of solutal concentrations obtained from theories. Specimens of differing compositions will be compared to equivalent layered foils to verify the molar volume effects in the model.

XTM offers new insight into the kinetics of the solute boundary layer during solidification. We thus have a strong desire to quantify the solutal gradients in the liquid ahead of the interface since this is a powerful tool in furthering the fundamental understanding of the solidification process. No one, to our knowledge, has a better means of measuring these concentrations. As a result, these details of absorption modeling are needed to best quantify the solute concentration measurements. To further this goal, it has been determined that a mechanism to measure absorption of a calibrated standard will need to be accommodated in the furnace design. This is because the measurement of solute concentration amounts to a measure of signal subtractions where there is normally no absolute reference point. The calibration standard will provide this reference.

8. Particle Pushing Observations

Experiments to measure particle pushing of zirconia particles in aluminum alloys were performed. To date, no evidence exists that verifies that pushing occurs in these systems. There was always the possibility that the solid phase wets the zirconia and would not lead to pushing but affirmations originating from Dr. Stefanescu's group in Tuscaloosa supported the pushing phenomenon. It was presumed that regardless of particle size, a sufficiently slow growth rate would lead to pushing. Rates down to 0.25 μm/sec were used with particles of 30 to 500 μm size in both alloys of and pure aluminum. Engulfment always occurred.

Examination of cylindrical Bridgman specimens prepared in Tuscaloosa similarly showed no evidence of the pushing phenomena. The XTM was used to determine the particle distribution both before and after unidirectional solidification. Post solidification examinations showed the particles to have moved but not necessarily to be pushed. The large (>400 μm) particles should have Stokes-settled in the ampoule during the prolonged melting/solidification cycle. A lack of settling prompted Dr. Kaukler to determine why particles seemed to be anchored both from gravity and from particle pushing. It soon became apparent that the casting quality of the samples including those for direct observation in XTM during solidification was inferior due to the large quantity of interior voids or porosity. In many samples, observed during melting and solidification in the XTM, voids developed throughout the process before solidification. This was a new phenomenon itself and was attributed to the different methods of specimen processing and/or the alloy composition.

The significant observation about voids is the frequent attachment of some porosity (perhaps an oxide veil) with particles or certainly with clusters of particles. Another observation was the lack of single particles regardless of particle size. The conclusion was

that there was oxide film formation on the surface of the melt which became trapped with particles when they were introduced to the melt. As a result, it seemed that few particles (if any) would be free. After unidirectional solidification, it seemed that many particles would be segregated to the outside surfaces of the specimens and few situated within the bulk of the metal. All of these observations showed an inherent difficulty with these systems.

In order to eliminate porosity from a casting with particles in it, an air melt of pure Al was made and particles introduced. It was difficult to blend the particles into the metal this way. Rods of this mixture were aspirated into glass tubes and examined. Few particles were in fact introduced and significant porosity was found within the specimens. The plan was to roll the rods to establish the thickness (1 mm) for the XTM solidification and weld the pores shut while shearing any oxide films away from the particles. This all seemed to work as planned but there no pushing.

After discussion about the problems with Dr. Stefanescu, another explanation was presented where there was some evidence that the slow growth rates needed for pushing raised the residence time of the particles with the melt in such a way to promote a reaction between the two materials. Such reaction products were not detectable by XTM but that doesn't eliminate the possibility. Evidence that residence time was an issue stemmed from an observation that pushing was detected in pure Al at high solidification rates (>35 μm/sec). This problem will be addressed by Dr. Stefanescu's group.

9. Presentations and Publications

Peter A. Curreri, and William F. Kaukler, "REAL-TIME X-RAY TRANSMISSION MICROSCOPY OF SOLIDIFYING AL-IN ALLOYS," to be published in Proceedings of the "Seventh International Symposium on Experimental Methods for Microgravity Materials Science Research," (presented at The Metallurgical and Materials Society Annual Meeting, Las Vegas, NV, Feb. 12-16, 1995). Also submitted to Metallurgical Transactions.

Gordon Conference on Gravitational Effects in Phisico-Chemical Systems.
Curreri and Kaukler. June 1995.