NASA-CR-197375

IN-74-CR

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X-Ray Microscope for Solidification Studies

Year End Report by William Kaukler, UAH

Introduction:

This report covers the second 6 month period for the year March 1, 1994 to February 28, 1995. The material outlined in this semi-annual report continues from the previous semi-annual report.

The Fein Focus Inc. x-ray source was delivered in September and coincides with the beginning of the second 6 month effort. As a result, and as outlined in the statement of work, this period was dedicated to the evaluation, testing and calibration of the x-ray source.

In addition, in this period the modeling effort was continued and extended by the Tiger series of Monte-Carlo simulation programs for photon and electron interactions with materials obtained from the Oak Ridge RISC Library. Some further calculations were also made with the absorption model.

Preliminary X-Ray Source Evaluation:

In a word, the performance of the x-ray source was superb. After installation and some fine tuning, the device was shown to be an excellent performer that in some ways exceeded advertised specifications.

Using the supplied tri-focus x-ray image intensifier, and the borrowed gold line resolution grid, it was ultimately possible to resolve the 2 micron lines on the grid in real time. It became clear that to achieve the highest resolutions possible from this instrument, limited by the intensifier, one needed to maintain the specimen placement within the first 4 to 5 mm nearest the source. The magnifications exceeded 500 X when specimens were placed with the nearest mm to the target. The grid was radiographed on Dupont 55 film and showed clearly the 2 micrometer lines. The clarity was so good that it can be assumed the resolution approaches 1 micrometer or better (with film). What was unexpected was that this resolution was available at the maximum power the machine would operate with. The input acceleration of 50 kV and 400 microamps offered 20 watts of input power. Due to internal computer imposed limitations, the power level drops off on either side of this point. The maximum power input was determined and is plotted in Figure 1. At this time, the reason for power drop off below 50 kV is not known. Above 50 kV, the 5 micrometer thick W target is protected by cutting the current as acceleration increases.

From prior communications with Fein Focus Inc. the power density of electrons onto the target was ostensibly determined by the limitations of damaging the W target given a limit to the removal of excess heat. The curves for the other machines they build approximately showed a linear relation with power level in watts and the same figure for the minimum focal spot size in micrometers (i.e. a 10 micron spot could tolerate 10 watts of power, and a 1 micron spot only 1 watt of power maximum). For the performance levels of resolution desired (sub-micron spot size), a low x-ray power would be expected. It seems that the electron optics of this High Definition tube

(NASA-CR-197375) X-RAY MICROSCOPE FOR SOLIDIFICATION STUDIES Semiannual Report, 1 Mar. 1994 - 28 Feb. 1995 (Alabama Univ.) 6 p

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significantly reduce the electron flux to the focus spot during the beam conditioning process. As a consequence, it has been observed that after prolonged operation (1 hour) at maximum power, the outer target surface was merely warm to the touch. Compare this to the uncomfortably high heat emanating from the other transmission tube target used for the feasibility studies not even operating at full power. This evidence supports the deduction that the power of the electron beam reaching the target is far less than that being generated at the filament.

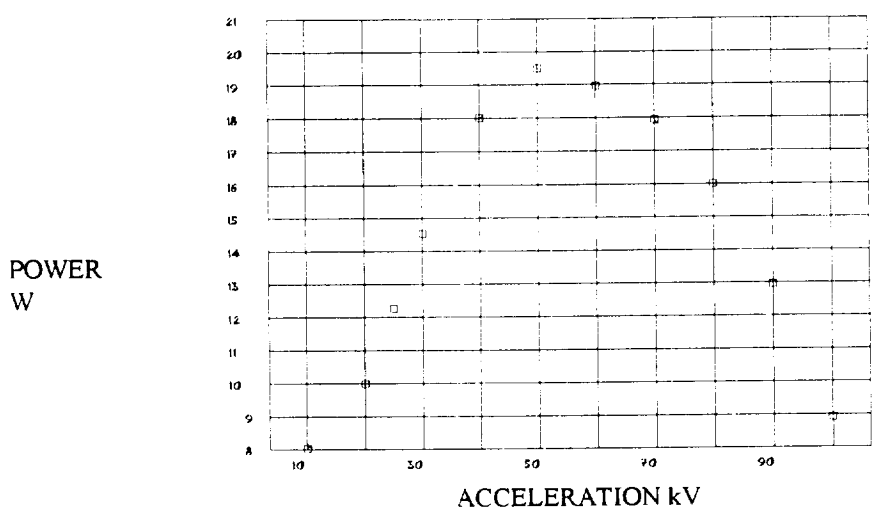

Figure 1. Fein Focus Inc. High Definition tube maximum power vs. electron acceleration over the full acceleration range.

Further experimenting showed that for real-time observation of specimens, acceleration voltages of 45 or at least 40 kV were needed. If the cooled CCD camera were used, the images could be detected down to 20 kV with significant increases in exposure time (several minutes). The low power output of this tube also limits observation in real time to thin metal specimens. The denser and thicker specimens require the higher voltages such as 80 or more kV for ease of observation.

A variety of specimen types were examined with interesting results. For example, real-time observation through 1 mm of tin or HgCdTe was not possible. Thin chips of BiSrCuO high T_c superconducting material were equally difficult to image. Thin sheet metal boxes made from steel, copper, brass or nickel did not prevent detection of the contents. Nickel beads of 30-100 micrometer diameter were easily imaged in real time and could be detected through 6 mm of Al. This showed the high contrast was available when such beads will be used for particle pushing experiments. Five mm diameter specimens grown in the ADSF furnace were examined. Iron and steel specimens did not reveal any interesting structures. However, old specimens of Al-In-Sn alloys were proving very interesting and the x-ray microscope views provided far more information than any metallography could ever have permitted. It seems the monotectic metal alloys, particularly Al-In and Al-Pb offer the most dramatic and informative microstructures.

Real-time observation of materials is truly informative. However, the best images were obtained when the cooled CCD camera was used where longer exposures, more gray level resolution and higher camera resolution produced such results that the x-ray intensifier was the limiting link in the imaging chain. The higher resolution and contrast offered by film radiography was explored with exemplary results. The near infinite depth of field the x-ray source offers permits views of materials few people have seen. The one micrometer (at the film) resolution of the radiographic film does not limit the image quality of any specimen in this instrument. Images as clear as those obtained from contact radiographic methods were obtained. Short exposures of a minute or so yielded the best results. However, good radiographs were taken using 15 or more minutes of exposure. However, the mechanical stability of the film, the specimen and the source sometimes was inferior to the source resolution and blurring occurs.

Measured Characteristics of Fein Focus Inc. High Definition Tube

Part of the characterization of the instrument carried out was the measurement of certain important variables that were not supplied by the Fein Focus Inc. people. Examples are output fluxes, magnification range, resolutions, power level and more as a function of acceleration voltage, current, and specimen to detector geometry.

A set of measurements were made to determine the magnification available. The measurements are compiled in a graph where the source and intensifier positions are fixed (as they always are). The calibrated wires and bars used to make the measurements were extremely precise and errors of a thousandth of an inch are the most one will see. The magnification as a function of distance from the target exterior onto the intensifier is plotted in Fig. 2.

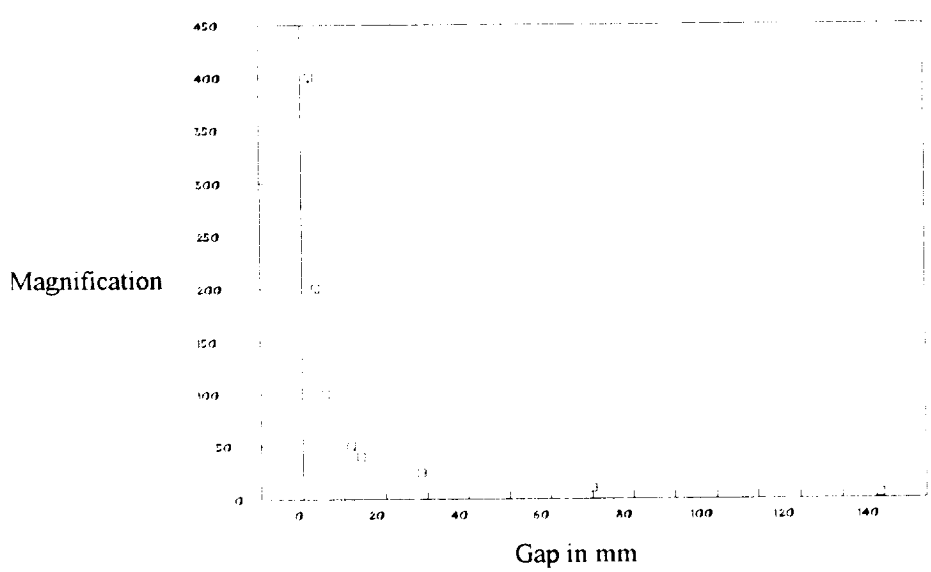

Figure 2. Magnification vs. distance from target surface.

As one can see, the significant magnification can only be achieved within the first 5 mm (1/4 of an inch) from the target. This is for the fixed distance from target to intensifier tube of 21 inches. The measurements could not be made higher than the graph shows although 800 X was achieved by another means. Shimming calibrated wires to the target surface provided the plot in Figure 3. The higher magnifications can be seen. The diameter of the wires themselves were in all cases accounted for in the distance calculations.

An important consequence is the high significance of the relative depth of the features within the specimen when the specimen is in this 5 mm zone. A 1 mm specimen, such as the kind anticipated for solidification studies can have a features displayed several hundred times different in magnification. This will not be as great a problem in the furnace since the furnace walls will impose a gap of at least 5 mm, some magnification difference will occur but not as unmanageably as when the specimen is magnified 400 X.

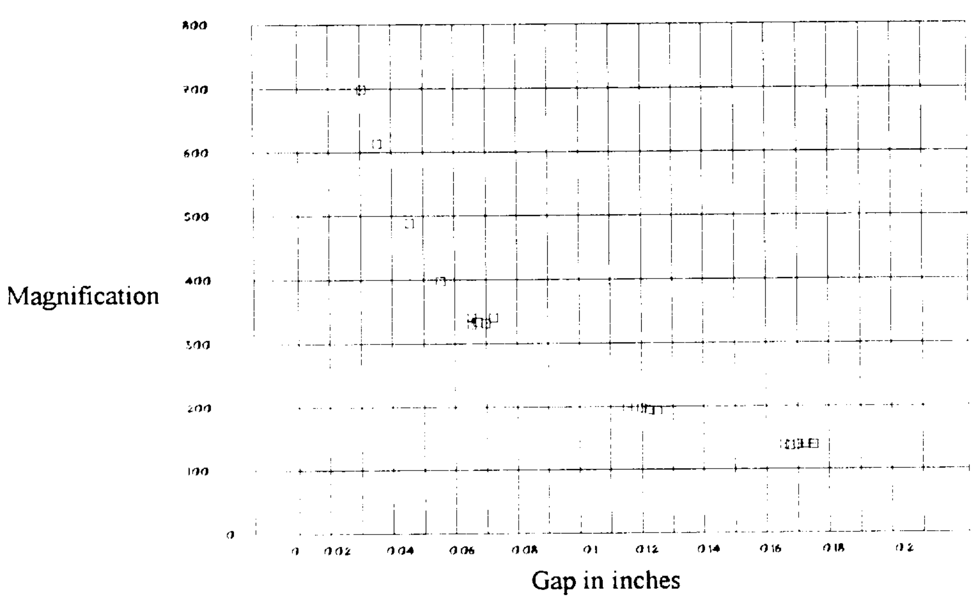

Figure 3. Magnification measured near the target surface.

Figure 4 is shown to compare the linearity of the measured magnification compared to a calculated one. Multiple measurements do show some scatter, but the line is near straight.

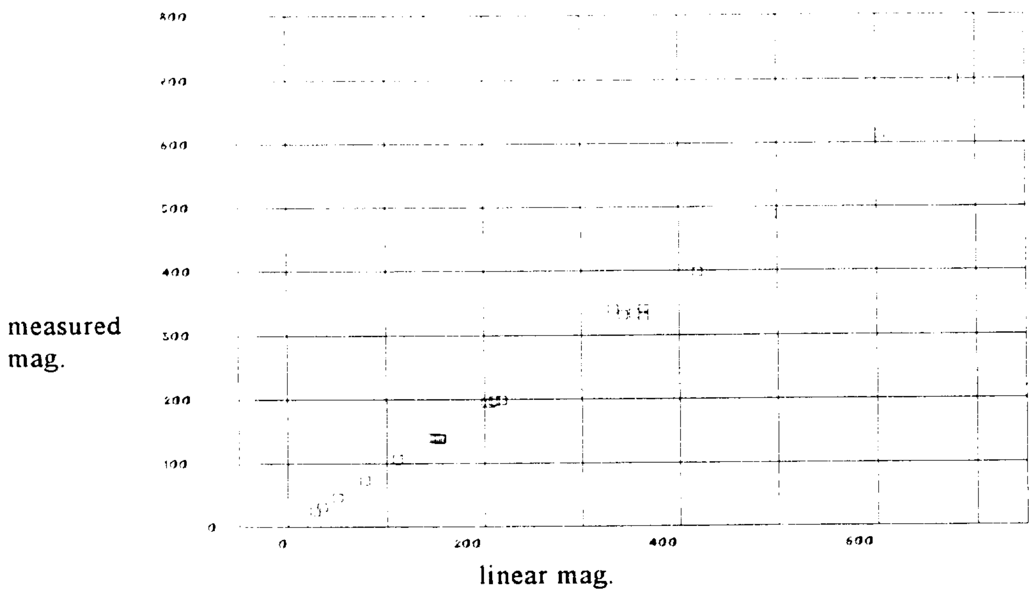

Figure 4. Measured magnification plotted against calculated magnification using similar triangles geometry.

The specimen table between the tube and intensifier can be moved down to provide 10 inches of workspace. The motors for this table are expensive and precise instruments. Each motor, although not set up for this, has installed an encoder which, if properly connected can be read and accurate table position obtained. The CNC option which would do this was not purchased but the encoders only need electronics to read them out. The joystick positioning control works effortlessly. However, at the high magnifications obtainable with the High Definition tube, the rate of minimum motion is still to high and positioning of a feature in the viewing screen is difficult. Some means of slowing this motion (in x and y only) is needed even at the expense of not being able to go as fast at the highest velocities.

A measure of the flux vs. input current was made for 30 and 50 kV using the ionization survey meter. The plots shown in Fig. 5 and 6 show a near linear dependence of output with current in the low ranges.

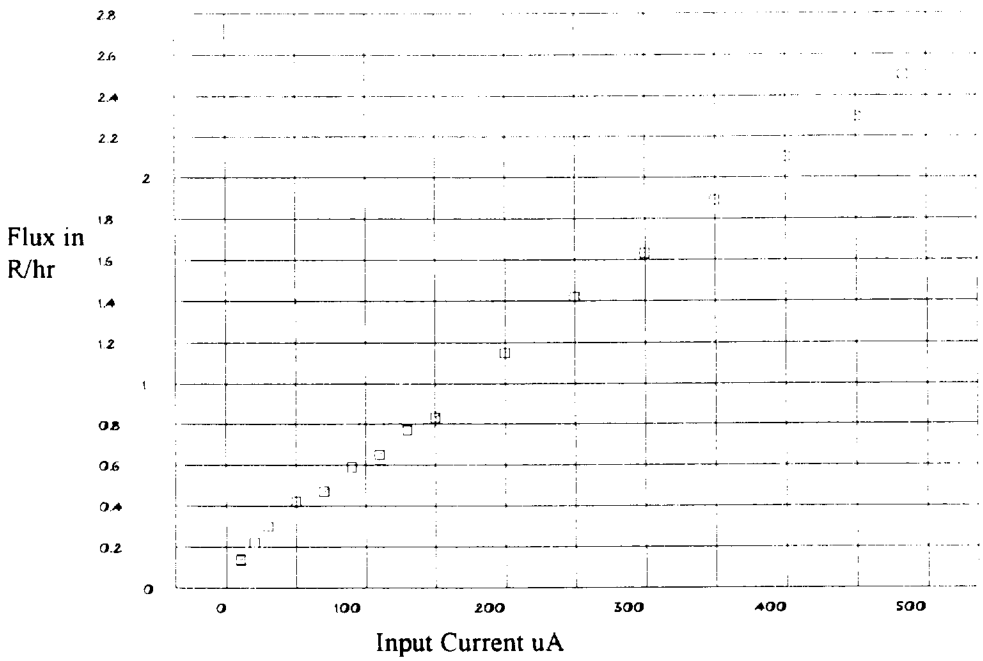

Figure 5. Measured flux vs. input current at 30 kV.

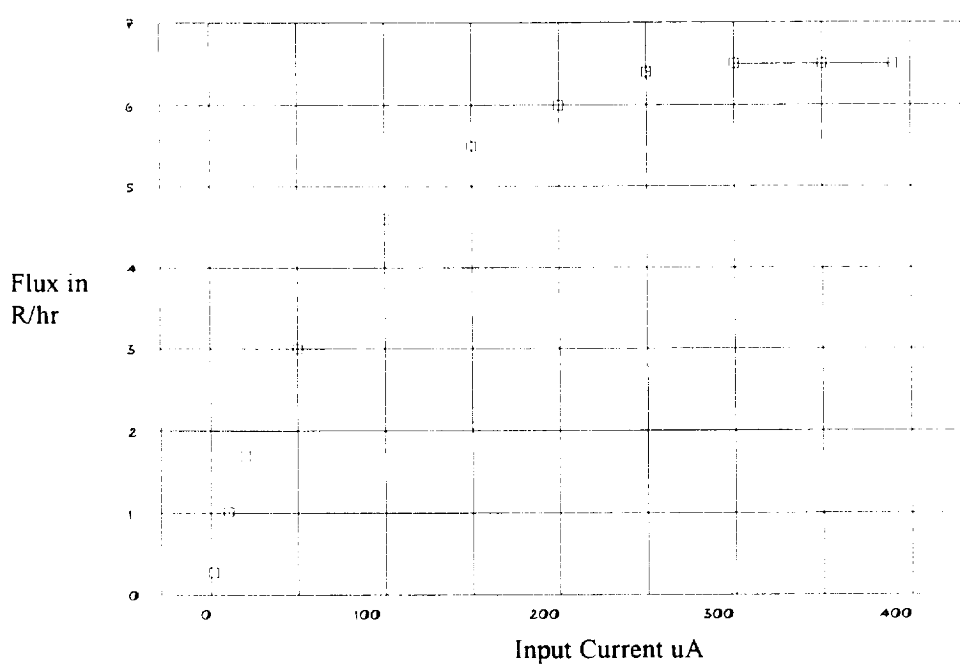

Figure 6. Measured flux vs. input current at 50 kV. The curve is caused by the meter becoming saturated at 6.5 R/hr.