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R.G. BLAKE
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ABSTRACT

This report contains the results of a detailed calibration of the JSEM-200 scanning transmission electron microscope operated with a magnetic specimen pole-piece as supplied by JEOL (Japan Electron Optics Laboratory) Ltd. The microscope, in this configuration, permits convenient examination of ferromagnetic specimens in the side-entry goniometer specimen stage with a point-to-point resolution better than 2.0 nm.

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CALIBRATION; ELECTRON MICROSCOPES; RESOLUTION; FERROMAGNETIC MATERIALS; MAGNETIC FIELDS

<u>CONTENTS</u>

1. INTRODUCTION

Ferromagnetic specimens can seriously reduce the resolution in electron microscope images because the magnetic field of the specimen distorts the magnetic field of the objective lens. This field distortion is asymmetric because the electron transparent area is seldom in the centre of the specimen. Such asymmetry deflects the transmitted beam from the optic axis of the objective lens with subsequent loss in image quality. Consequently, the incident beam must be tilted to obtain axial illumination. In practice, this becomes extremely tedious as the microscope requires re-alignment after each specimen shift. The problem is particularly severe in microscopes which are equipped with side-entry goniometer stages, such as the JSEM-200, because the objective is then an immersion lens. The continual microscope re-adjustments required during specimen tilting effectively defeat the usefulness of the side-entry goniometer for serious crystallographic studies of ferromagnetic specimens.

Fortunately, the problem of beam misalignment during specimen tilting has been overcome recently by Shirota et al. (1976) who developed a new lens system for use in the JSEM-200. Essentially, the standard objective lens pole-piece is replaced by a new pole-piece which gives a reduction by a factor of about 1200 in the magnetic field strength at the specimen positions in the normal side-entry goniometer. The pole-piece was purchased from JEOL (Japan Electron Optics Laboratory) Ltd. It was supplied with a matched projector lens pole-piece but initial experiments revealed that the maximum magnification of the microscope was limited to about 35,000 times. Pairing of the magnetic specimen objective lens pole-piece with the standard projector lens gave a more useful magnification range without any detectable loss of performance. Hence the projector pole-piece supplied with the low magnetic objective lens pole-piece was discarded. The installation required changes in the transformer tapping on the lens supply circuit and minor electrical adjustments to ensure a suitable range of objective lens currents for image focusing. These circuit modifications were made so that, apart from the transformer tapping, the objective lens circuits could be changed back to standard settings by a single switch. This flexibility in change-over from magnetic specimen to standard objective lens pole-piece is desirable because the standard lens is essential for the operation of the microscope in the STEM mode (Scanning Transmission Electron Microscope).

Generally, the change-over requires 2-3 hours. The standard lens also possesses superior optical characteristics for non-magnetic specimen applications.

This report describes the results of calibration measurements on the AAEC JSEM-200 electron microscope operated at 200 kV with the magnetic specimen pole-piece. Results of calibrations with the standard objective lens have been published previously by Blake, Jostsons and Kelly (1975).

Note that the various lens currents on the JSEM-200 are displayed on a milliammeter via a selector switch. To overcome parallax associated with reading of the meter, a digital voltmeter has been installed in our microscope. Consequently, the actual readings in the calibration are measurements of the voltage across the shunt of the milliammeter. The relationship between the milliammeter current and the digital voltmeter reading is 1 mA ≡ 2 mV.

2. MAGNIFICATION CALIBRATION

The magnification calibration was carried out on ranges M2 and M3 of the microscope, the useful range of magnification available. The magnification is mainly a function of the intermediate lens current. However, variations in specimen height can also affect the magnification and so the objective lens current must be monitored as well. Changes in specimen height were achieved by raising or lowering the goniometer stage via the 'Z' control. A replicated grating with a spacing of 2160 lines per mm was used to calibrate the magnification. The results are shown in Figure 1 and the necessary lens settings for selected magnifications are shown in Figure 2.

3. CAMERA CONSTANT

For small diffraction angles, Bragg's Law can be written as

$$Rd = \lambda L \quad , \qquad \ldots(1)$$

where R is the radial distance between the diffracted and transmitted beams on the photographic plate, d is the interplanar spacing, λ is the electron wave length and L is the camera length. The camera constant λL was determined as a function of objective lens current (Figure 3) from measurements of the diffraction rings from an evaporated, fine grained gold film (a = 40.79 nm). These measurements were made with the selected area diffraction selector switch set in position 2.

4. ROTATION CALIBRATION

It is essential to be able to transfer crystallographic information from the diffraction pattern to the image. In most electron microscopes a rotation between the object and image is produced by the magnetic lenses. The magnitude of the rotation depends on lens currents. This rotation must be calibrated relative to the diffraction pattern. The latter is always produced with constant lens excitations.

The rotation calibrations were carried out using αMoO_3 single crystals. The results, shown in Figure 4, include the 180° Groves and Whelan (1962) inversion which is determined by the method described by Head et al. (1973). All measurements refer to photographic plates viewed with the emulsion side up. To avoid ambiguity, the variation of the rotation with lens current is shown pictorially in Figure 4 by a symbol with one-fold rotational symmetry.

5. RESOLUTION

The point-to-point resolution of the JSEM-200 using the standard objective lens is approximately 0.7 nm. The magnetic specimen objective lens pole-piece employing low flux density at the specimen is expected to have a considerably higher coefficient of spherical aberration (Hirsch et al. 1965) and thus a poorer resolution. From images of gold-palladium particles evaporated on a thin carbon film the resolution is estimated to be better than 2.0 nm. Similar resolutions have been observed in thin specimens of iron alloys.

6. SUMMARY

The magnetic specimen pole-piece gives an acceptable resolution and range of magnification in the JSEM-200. Its advantage is that it enables disc specimens of α-Fe to be used in the side-entry goniometer stage which can be manipulated over the full design range eliminating the need for significant corrections to the electron beam illumination system.

The calibrations correspond to a current value of 53.3 μA in the jack of the reference battery box. To obtain reproducible results, this current must be checked periodically and the reference batteries replaced at the first sign of decrease in the test jack current. Otherwise the calibrations may change by as much as 25 per cent.

7. REFERENCES

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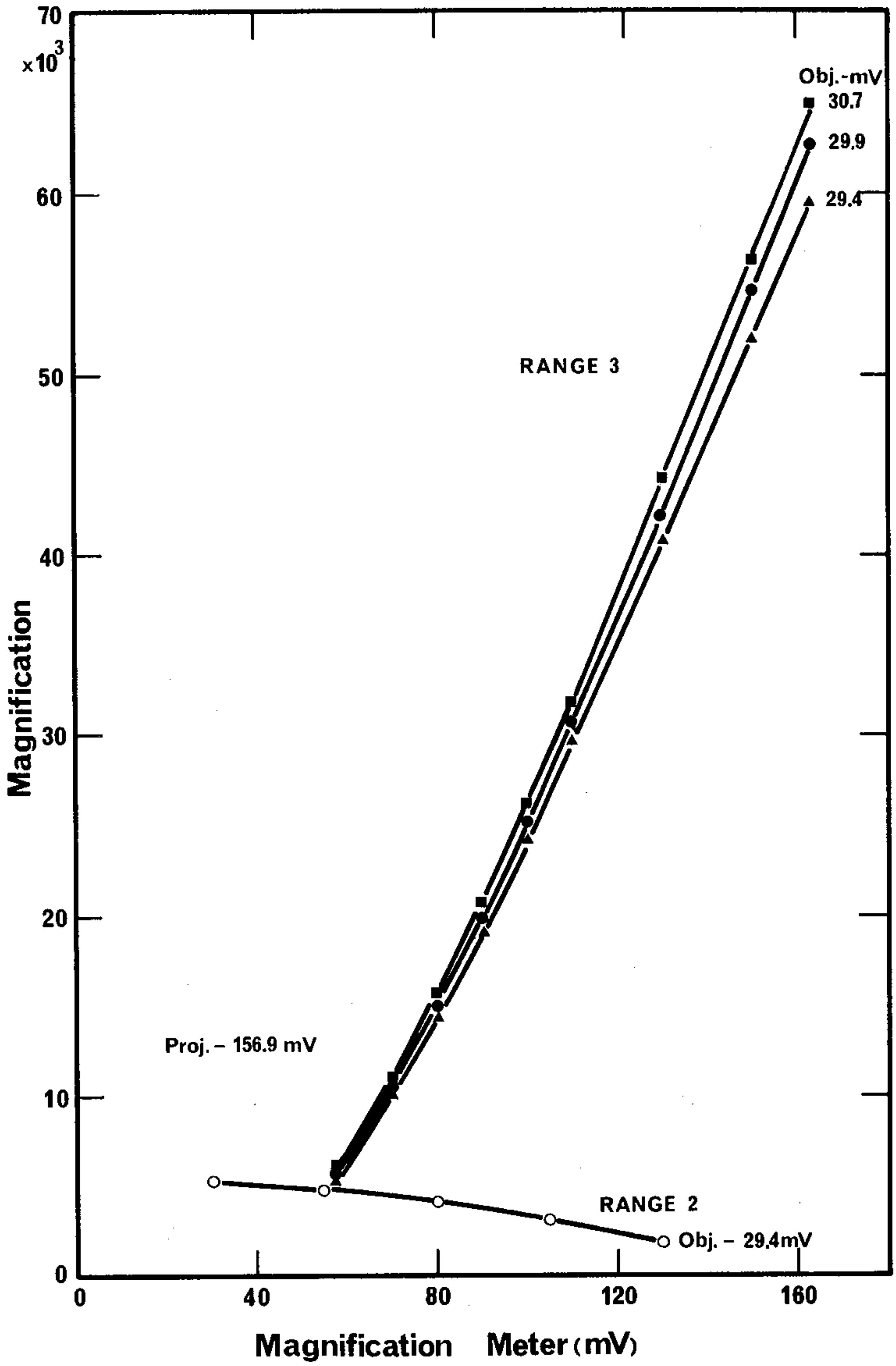

FIGURE 1. MAGNIFICATION CALIBRATION FOR 200 kV AS A FUNCTION OF MAGNIFICATION METER READING AND OBJECTIVE LENS EXCITATION

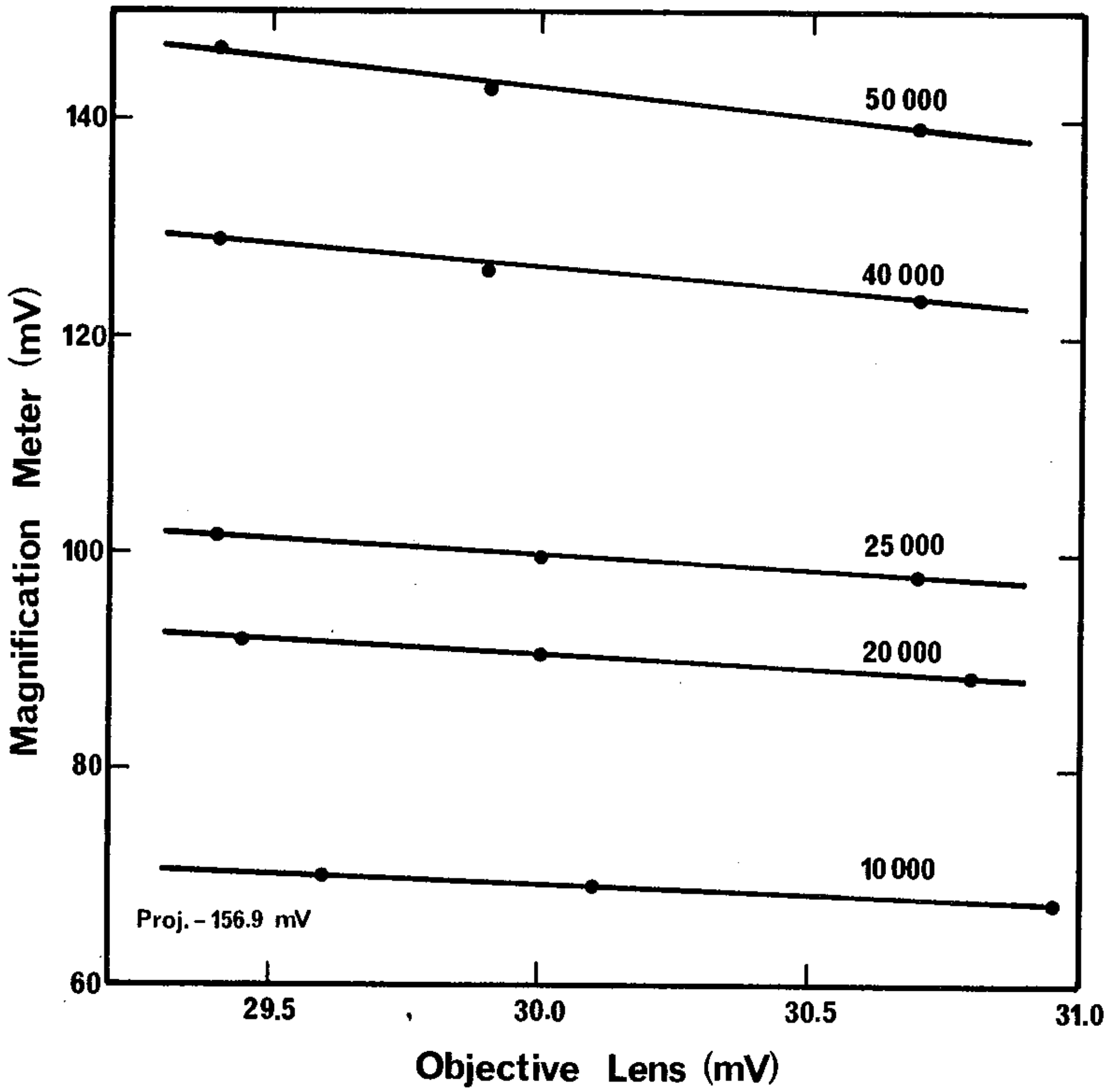

FIGURE 2. THE RELATIONSHIP BETWEEN OBJECTIVE LENS AND MAGNIFICATION METER READINGS AT SELECTED MAGNIFICATIONS FOR RANGE 3 AT 200 kV.

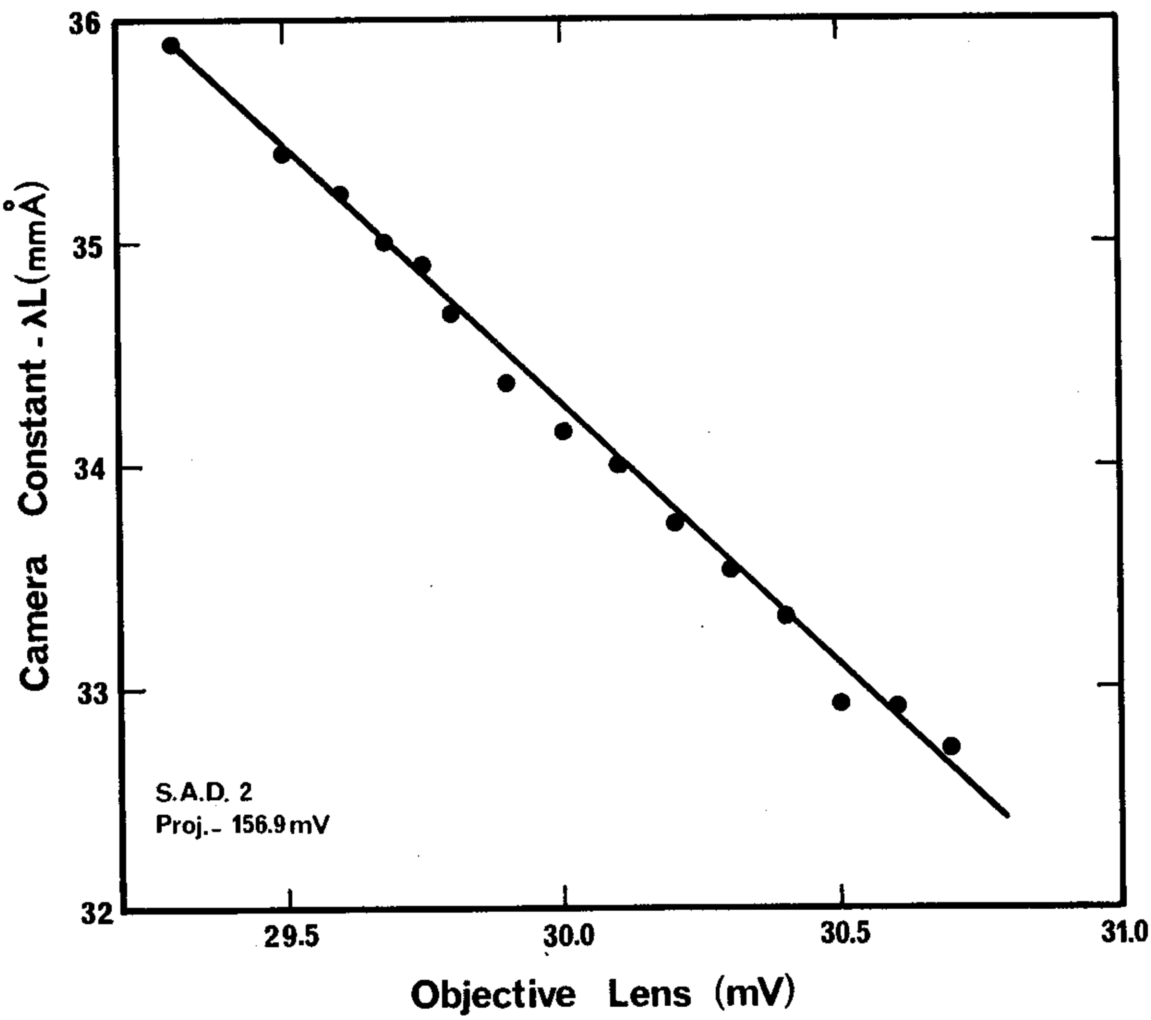

FIGURE 3. VARIATION OF CAMERA CONSTANT, λL, WITH OBJECTIVE LENS 'CURRENT' FOR S.A.D. 2 POSITION AT 200 kV.

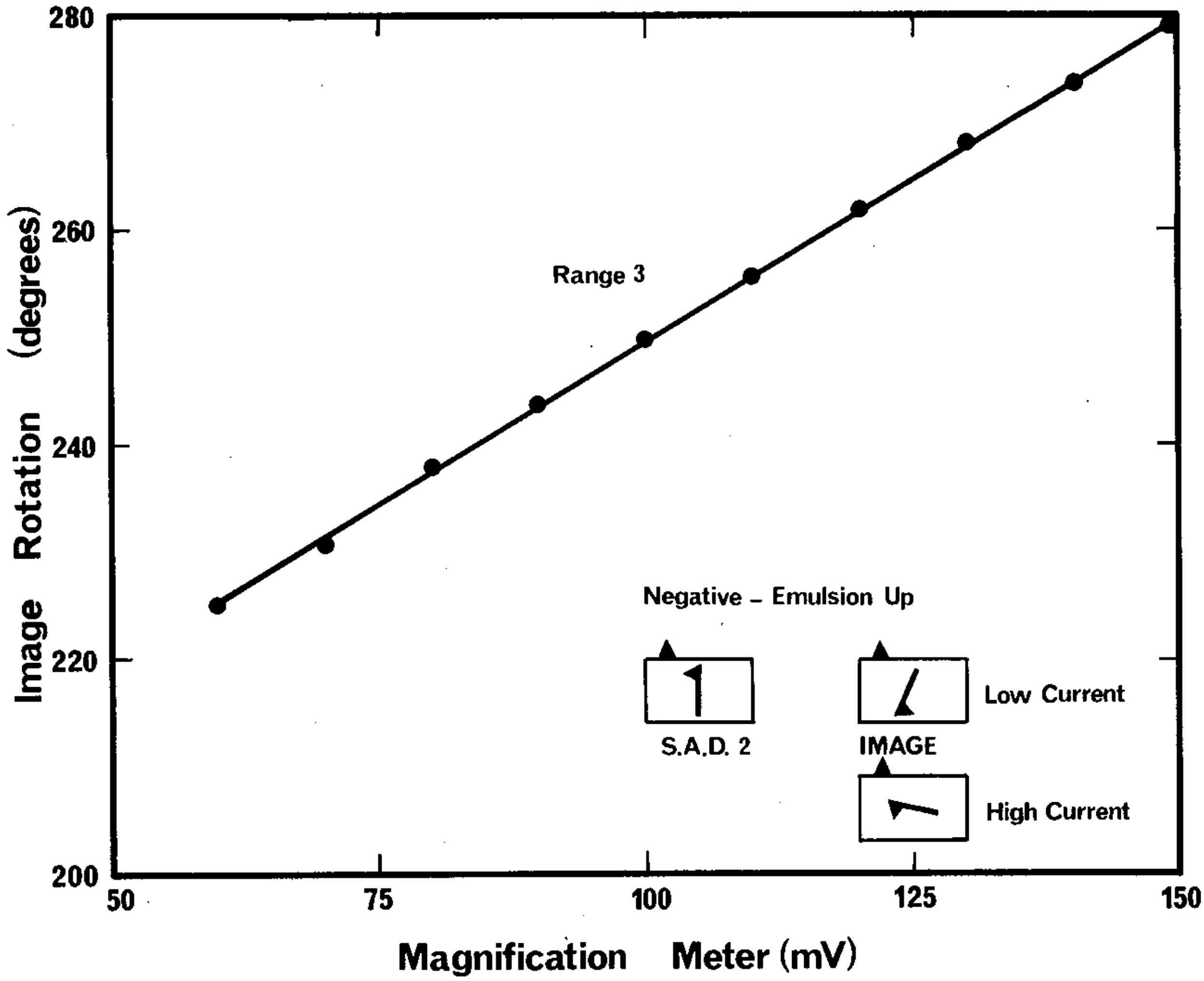

FIGURE 4. RELATIVE ROTATION OF THE IMAGE WITH RESPECT TO THE DIFFRACTION PATTERN (AT S.A.D. 2) AS A FUNCTION OF MAGNIFICATION METER READING AT 200 kV FOR RANGE 3.