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6Ni STEEL BY STEREO SEM TECHNIQUE

Gianni O. Fior
(M.S. thesis)

June 1982

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M.S. Thesis

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This work was supported by the Director, Office of Energy Research, Office of Development and Technology, Magnetic Systems Division of the U.S. Department of Energy under contract number DE-AC03-76SF00098.

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ABSTRACT

Systematic three dimensional (3D) quantitative analyses of fracture surface on 6Ni cryogenic steel are used to study the effect of temperature variations on the geometrical factors that describe the shape of ductile dimples (DD) and quasicleavage (QC) facets. Stereo SEM techniques combined with stereo photogrammetry provide the tools to perform such analysis on standard Charpy specimens tested with appropriate instrumentation over a 300 degrees temperature range. Mechanical properties and tridimensional geometrical factors are found to be intimately related. The influence of temperature on DD and QC is considerably different, showing that quasicleavage is a transitional fracture mechanism that serves as a link between ductile and cleavage fracture.

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GIANNI O. FIOR

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1. INTRODUCTION

Fracture of materials has been a major concern of man since he began to make tools and to build structures which because of fracture eventually failed to function. More than four hundred years ago Leonardo da Vinci pioneered fracture analysis by designing and conducting tensile tests of iron wires and recording information on strength and fracture location.^[1] Today's development of new materials to meet innovative technological applications demands knowledge of the complex processes of fracture that take place under varied testing conditions. This is of particular importance in the development of materials for use at a wide range of temperatures. In the case of cryogenic steel for example, a change in mechanical behavior due to temperature is also accompanied by a change in the fracture appearance. Therefore a thorough investigation of the mechanisms of fracture can provide information to design or improve modern alloys.

In general, fracture of polycrystalline materials can be classified according to crack path into two main categories:

- i) Transgranular fracture: occurs across grains and grain boundaries.
- ii) Intergranular fracture: occurs between grains and along grain boundaries.

Within the transgranular fracture regime, several mechanisms have been established:^[2,3]

- Ductile Dimple:** microvoid nucleation, growth and coalescence leading to highly deformed fracture surfaces and associated with high fracture energy.
- Cleavage:** separation of preferred crystallographic planes leading to undeformed fracture surfaces and associated with very low fracture energy.
- Quasicleavage:** a mixture of the ductile dimple and cleavage fracture modes. Like dimples its origin is strain related but with considerably less deformation present in the fracture surface, which looks mostly flat, like in cleavage but without the *river patterns* or *tongues*. It is usually present on the fracture surface of quenched and tempered steels tested at low temperature, below the ductile to brittle transition temperature (*DBTT*). It is associated with low fracture energy.

Using optical and electron microscopy, a study of these fracture modes can qualitatively correlate them with the mechanical behavior of the materials. However, a clear understanding of these fracture mechanisms demands more than simple qualitative correlation. Quantitative fractographic analysis, on the other hand, has been normally limited to only two dimensions (*2D*), leaving out important information such as depth, roughness, inclination, etc. The use of stereo techniques to obtain three dimensional (*3D*) qualitative and quantitative information of fracture surfaces extends the perception of the real fracture appearance, and also increases the accuracy of the quantitative analysis by introducing at least one third more information than that obtained in *2D*.

Nevertheless, *3D* fractographic analysis also presents limitations. The efficiency in the retrieval of large amounts of measurements is lower than in most *2D* standard quantitative metallographic methods applied to fracture analysis. This drawback can be significantly alleviated by automation of the *3D* measuring process.^[4,5] By studying the various fracture mechanisms through analyzing features on the fracture surface, it becomes clear that any result from such an investigation will be a statistical inference from a limited sample of an extraordinarily large population of features. Careful sampling of fracture surfaces must be undertaken for

unbiased and meaningful information that could be used in cryogenic materials development. A 3D quantitative fractographic analysis of cryogenic steel is used in this work to characterize the fracture modes over a wide range of testing temperatures.

2. EXPERIMENTAL PROCEDURE

2.1. MATERIAL AND HEAT TREATMENT

The material used for this study was a commercial 6Ni steel obtained from the *Nippon Steel Corporation* in the form of a one-inch thick plate. The chemical composition (in weight %) of this alloy was as follows: $C=.063$; $Mn=1.21$; $Si=.29$; $P=.008$; $S=.01$; $Ni=5.86$; $Cr=.69$; $Mo=.20$ $Fe=balance$. This alloy is designed for cryogenic applications since it maintains its high strength and high toughness at low temperatures. Normally this alloy is given the conventional quenching and L-tempering (*QLT*) heat treatment^[6] or the two B-tempering (*2BT*) treatment^[7-9] in order to depress the *DBTT* to even lower temperatures. For this work, however, easy access to testing temperatures below and above the *DBTT* was desired. Therefore, the material was annealed at 1000 °C for two hours to remove any previous thermomechanical treatment and then water quenched (*WQ*) to raise the *DBTT* above liquid nitrogen temperature.

2.2. MECHANICAL TEST

Standard V-notch Charpy specimens (figure 1) were cut from the *WQ* plate with the longitudinal axis parallel to the rolling direction and the loading direction perpendicular to it. Three or more specimens were tested at each temperature, varying from 77 K to 373 K. Subzero temperatures were obtained using different proportions of liquid nitrogen, isopentane (2-methyl butane) and methyl alcohol. Charpy impact tests at subzero temperatures were performed following standard procedures of *ASTM #23-72*. The tests were conducted in a

Tinius Olsen M64 Charpy machine, instrumented with a *DYNATUP 500* system to monitor the dynamic behavior and record both load and absorbed energy at any time during the test. The hammer striking velocity was 512 cm s^{-1} for all tests, and the procedure used for the instrumented Charpy tests followed the recently proposed standard method.^[10] Upon calibration of the instrumentation, a very good agreement was always obtained between the absorbed energy measured from the dial and from the oscilloscope display. Figure 2 shows an idealized and an actual load versus time curve.

2.3. STEREO SCANNING ELECTRON MICROSCOPY

The specimens fracture surfaces were directly observed using the backscattered electrons in the the *SEM*. A special sample holder, designed and built by this author, was used to position the full size specimens always with their fracture surface normal to the incident electron beam. Magnification was calibrated for *X* and *Y* axes and was checked before each session with a standard calibration grid. The *SEM* used had an eucentric[†] specimen tilt stage with $\pm .5$ degree accuracy. Stereo pairs of fractographs were taken using the specimen tilting method.^[11] The specimen was tilted symmetrically across the flat surface to minimize distortion of the images and to simplify 3D equations by using the central plane image equal to zero tilt.^[12,13] The optimum total tilt angle was experimentally determined to be 10 degrees for the given characteristics of the stereoscope focal length, material topographic configuration and operator interpupillary distance.^[14] Magnification was held constant at 2000 *X*, which allowed for enough resolution of all present features and permitted the use of the parallel projection approximation for the extraction of 3D information.^[15,16]

[†]Eucentric is the generally accepted term used to describe a specimen stage in which the tilt axis, rotation axis and optical axis intersect each other in one point, allowing for specimen tilt and rotation without the need for readjusting either the specimen position or the focus.

2.4. STEREO MEASUREMENTS

Stereo pairs of *SEM* fractographs were observed under a *WILD ST4* mirror stereoscope. As mentioned in the introduction, the process of taking 3*D* measurements can be very slow if done manually using a parallax bar. In this work a 1224 *Numonics* digitizer was used to obtain all parallaxes (difference in length between two points as they are recorded in different tilted photographs) and other measurements from the fractographs. Digital display and printed record of the measurements were automatically produced at the same time the data was stored in a terminal bubble memory through a digitizer-terminal interface. Later, data groups were sent via telephone line to the central computer facility where a program in *BASIC* written by this author processed the information to generate all 3*D* parameters, statistical analyses and graphics. Figure 3 shows a flow chart of this measuring process.

Three dimensional measurements do not need to be taken while perceiving a 3*D* illusion *stereopsis*.^[17] However, in this work all 3*D* and 2*D* measurements were taken by stereo visualization of the fractographs to produce a better qualitative depth correlation of the 3*D* features being measured as well as a superior accuracy in the parallax measurements.^[12] A simple scheme of the geometrical set up in the *SEM* for the specimen tilt method, assuming parallel projection, is shown in figure 4.

2.5. SAMPLING

The 2*D* appearance of Charpy impact specimens at four different testing temperatures as seen in the *SEM* are shown in figure 5. From these fractographs, two results are noteworthy. First there is a definite change of predominant fracture mode, and second, there are an infinite number of different shapes of features present within each fracture mode. In order to classify these features, a very restricted definition was used so that no subjective judgement was required from the operator. The definition specified in this work divides the features in two categories:

Ductile Dimples DD: a void of concave shape with a protuberant edge or border ridge such that if starting from an initial point P one may follow it in any direction and return back to the same point P, circumscribing the void completely and without interruptions. Such edges are clearly observable in *SEM* fractographs given the higher contrast produced by the pronounced height also known as the *edge effect*.^[18]

Quasicleavage Facets QC: features that fail to be classified as DD will be considered QC facets for the purpose of this work.

Figure 6 illustrates some examples of DD and QC according with the definition given above.

For each sampled feature a total of four data numbers were obtained:

i) Class DD or QC (dummy variable): this data was obtained through the stereoscope by the operator using the adopted convention and recorded via the keyboard into the terminal's bubble memory.

ii) Projected Area *A*: defined as the area circumscribed by the edge or ridge of the feature. It was directly measured with the digitizer using the stereoscope and corrected later by the topographic curvature angle.

iii) Maximum Depth *Z*: defined as the difference in height between the top and the bottom of the center of the feature. This parameter was calculated from the measurement of the parallaxes taken with the digitizer and stereoscope.

iv) Topographic Curvature: defined as the angle of inclination of the feature with respect to the normal to the fracture surface (also referred to as the central plane). This parameter is also calculated from parallaxes measured with the digitizer and stereoscope.

Figure 7 shows an idealized fracture profile with the 3D parameters as measured in this work.

The relation between the difference in height or elevation and the parallax measurements taken with the digitizer are given by the following equation (see figure 4):

$$Z_c = \frac{D_r - D_l}{2 \times \sin \alpha} = \frac{P}{2 \times \sin \alpha} \quad (1)$$

where:

Z_c = difference in height referred to the central plane

P = parallax value

α = half of the total tilt angle between left and right fractographs

Introducing the magnification M of the fractographs (1) becomes:

$$Z_c = \frac{P}{2 \times M \times \sin \alpha} = \frac{P}{k} \quad (2)$$

where:

$k = 2 \times M \times \sin \alpha$ = constant throughout this work

The above equations are derived assuming parallel projection ($M > 500$) [11-14,16,19]

Even with the automation implemented for the measuring process, the number of features makes it virtually impossible to sample the whole fracture surface of the specimens at the magnification selected (2000X). Figure 8 shows an idealized Charpy specimen fracture surface, in which only a small central area S , with relatively stable crack propagation, was used for sampling. At the magnification used, a total of 2,400 fractographs would be necessary to completely sample such an area for just one specimen. Instead, a random sampling technique was used, in which both the stereo fractographs and the features measured were selected after computer generated random coordinates were obtained for each specimen. Twelve random features were measured for each of six different stereo pairs of fractographs randomly taken within the central area S of each specimen at six different testing temperatures. Figure 9 shows a sampling scheme for one testing temperature.

3. RESULTS

3.1. MECHANICAL PROPERTIES

As desired and expected from the material heat treatment combination, the Charpy impact tests showed a gradual ductile to brittle transition of the total absorbed energy measured with the instrumented tup. As shown in figure 2a, the load-time history of these tests can also provide a good estimate of crack nucleation and propagation energies.^[20,21] Figure 10 summarizes the information obtained from the instrumented Charpy tests over a 300 degrees temperature range. Defining the *DBTT* as the temperature corresponding to the midpoint between the upper and lower shelves, it is found that for both total absorbed energy E_a and estimated crack propagation energy E_p , the *DBTT* was approximately 150 K (see figure 10). The parameters E_a and E_p also show a wide transition region. The estimated crack nucleation energy E_n instead has a narrower transition region with a very well defined upper shelf that drops at about 175 K to give a *DBTT* of 125 K. In the brittle region it was observed that E_n accounts for more than 90% of the total energy absorbed by the specimens. An interesting result from the instrumented Charpy data is provided by the plot E_n vs. T (figure 10) which shows that the fracture nucleation mechanism is virtually the same down to 170 K. This result of the mechanical behavior, if correct, should also be found in the proposed fracture analysis.

3.2. FRACTURE SURFACE ANALYSIS

3.2.1. Projected Area A :

The average projected area A of the features sampled in the fracture surface was found to decrease with increasing testing temperatures (figure 11). The plot $1/A$ vs. T shows a *DBT* shape with a transition temperature of 170 K. The same characteristics can be observed from a frequency distribution diagram of A for all features at six different temperatures as shown in figure 12. A more detailed observation can be made from the frequency distribution diagrams

for ductile dimples *DD* and quasicleavage facets *QC* presented in figures 13 and 14 respectively. It is important to note that while *QC* histograms show a shift of the density function toward higher areas at lower temperatures, the *DD* histogram remained unshifted and maintaining the same shape. These results suggest that the *DD* projected area is not affected by changes in testing temperature.

3.2.2. Maximum Depth *Z*:

The average maximum depth *Z* for all the features was found to increase with increasing testing temperature. The plot of *Z* vs. *T* also follows a *DBT* shape with a transition temperature of 170 K (see figure 15). By similar analysis to that used for *A* above, the *Z* parameter behavior can also be confirmed by the histograms for all features shown in figure 16. Again the change of distributions for *QC* contrasts with the constant distributions of *DD* (see figures 17 and 18).

3.2.3. Proportionality Factor *V*:

Given the characteristics of projected area *A* and maximum depth *Z* found in this work, it seems appropriate to define a proportionality factor *V* that incorporates both geometrical parameters *A* and *Z*. *V* was defined as the ratio of *Z* and the square root of *A*

$$V = \frac{Z}{\sqrt{A}} \quad (3)$$

Note that *V* is dimensionless and it represents a shape factor or an aspect ratio for the fracture features. This factor contains all the 3D geometrical information that was measured from the fracture surface. The plot of the calculated *V* vs. *T* is given in figure 19. Its *DBT* shape can be verified using the histogram shown in figure 20. Separate curves of *V* for *DD* and *QC* are plotted in figure 21 and their histograms are presented in figures 22 and 23. The plot of *V_{DD}* does not show any dependence on temperature, while for *V_{QC}* there is a very smooth *DBT* region with a *DBTT* at about 175 K. Other important results from this graph are the maximum

and minimum value of V_{QC} . The upper limit just below and almost tangent to the constant V_{DD} value, suggests that geometrical similarities exist between DD and QC . The lower limit instead, is set at a value greater than zero suggesting the existence of other metallurgical limiting factors for the minimum value of V_{QC} .

3.2.4. Density of Ductile Dimples ρ :

This variable, defined as the number of ductile dimples per unit of sampled area, shows a very drastic and narrow DBT with a transition temperature around 175 K (figure 24). The combination of this result with the constant geometrical V_{DD} factor indicates that the effect of low temperature is to inhibit the nucleation of DD rather than changing their shape.

3.2.5. Roughness Factor F :

The creation of at least two new surfaces is inherent to the process of fracture. A considerable portion of the total absorbed energy is consumed by the generation of these free surfaces and therefore some relationship between the energy absorbed and total surface area must exist. One way of studying this relationship is by introducing a roughness factor F defined as the ratio of the real total area of topographic fracture and its corresponding specimen cross sectional area. The real total area of topographic fracture is estimated as the sum of the measured projected area A plus the area of the feature wall defined as the maximum depth Z times the perimeter of the feature which for this purpose is assumed of circular shape:

$$F = \frac{\sum_{i=1}^n (A_i + (Z_i \times B_i))}{S} \quad (4)$$

where:

F = roughness factor (dimensionless)

A_i = projected area of the i th feature (μm^2)

Z_i = maximum depth of the i th feature (μm)

B_i = perimeter of the i th feature (μm)

S = specimen's sampled cross section (μm^2)

Figure 25 shows the graph of F vs. T . Here there is a *DBT* with similar characteristics to those obtained from other 3D parameters. However, the physical interpretation of F is of greater significance since it contains, by definition, all previously discussed 3D parameters and it is directly related to the energy required for fracture to occur.

3.3. STATISTICAL ANALYSIS

3.3.1. Confidence Level:

As seen in figure 8, an exhaustive quantitative fractographic analysis of all the fracture surface is impractical and virtually impossible. Therefore only a random sample of size 72 was chosen for each temperature (figure 9) in order to obtain the information necessary to make statistical inferences about the behavior of the population of features present on the fracture surface. The results from such analysis ($A, Z, V, etc.$) however, are only estimates of the *true* unknown values. The uncertainty of any experimental parameter can not be avoided; it can be either ignored or measured. In this work, a parametric statistical method was used to find the level of confidence at which the results were obtained.^[22]

In classical statistics, the level of confidence $X(\%)$ that the *true* unknown value of a variable Y , is within a given interval $[Y1, Y2]$ centered at $\bar{Y}$ (the estimated value of Y) can be found in the following way:

$$Y = \bar{Y} \pm \Delta \bar{Y} \quad (5)$$

and

$$\Delta \bar{Y} = \frac{t_{p,q} \times s}{\sqrt{n}} \quad (6)$$

where:

$\Delta \bar{Y}$ = half interval size (symmetric case) = $(Y_2 - Y_1)/2$

t = critical point (t -distribution)

s = sample standard deviation

n = sample size

p = probability that $Y \geq Y_2$

q = degrees of freedom = $n - 1$

solving for t :

$$t_{p,q} = \frac{\Delta \bar{Y} \times \sqrt{n}}{s} \quad (7)$$

The value obtained for $t_{p,q}$ can be used then to calculate p from the t -distribution density function and to find the level of confidence X defined as:

$$X(\%) = (1 - 2 \times p) \times 100 \quad (8)$$

Three levels of confidence corresponding to three different interval sizes were found for the projected area A , maximum depth Z and proportionality factor V at each of the six sampled temperatures. For each interval size, the average of the confidence levels over all the different temperatures is graphically shown in figure 26. From these graphs two general results can be observed. First, the confidence level for V is always greater than for A or Z alone, which implies that their combination into a single parameter strengthens the confidence level. Second, as expected from the definition of DD used here, the confidence level in decreasing order is $DD > ALL FEATURES > QC$.

A confidence level was also found for the regression line of V_{DD} , which in figure 21 is shown to be a constant independent of T . The confidence level is 97% for the intercept $\pm 10\%$ interval size and 99.6% for $\pm 20\%$ interval size. These are very high confidence levels for an experimental parameter of this kind.

3.3.2. Nonparametric Tests:

All the previous classic statistical analysis has been based on two fundamental assumptions: *independent* random variables (observations and measurements), and *normal* distribution of population. The former and perhaps the most crucial assumption is supported by the fact that all coordinates of the sampled features as well as those of the stereo fractographs were randomly generated by a computer program for each temperature. Nevertheless, this assumption was verified by two different nonparametric methods: the *Runs* test and the *Mean Squared Successive Difference* test.^[23] Both methods satisfactorily confirmed the randomness of the sample variables.

The normality assumption if wrongly used, could severely affect the confidence level validity. This can be indirectly detected by using a nonparametric *Sign* test on the population median and comparing the confidence level for same size intervals with the confidence level for the population mean. If greater confidence levels can be achieved through the non-parametric test, it is generally implied that the normality assumption was violated. In all cases tested, the confidence levels for the median were similar or lower than those for the mean which indicates that the normal population assumption could be used as first approximation. Other distributions (e.g., Weibull) are suggested for more detailed statistical analyses.

4. DISCUSSION

4.1. MECHANICAL PROPERTIES AND FRACTURE APPEARANCE

Throughout the experimental results of the 3D quantitative analysis a close relationship was consistently identified between the 3D geometrical parameters and the mechanical properties of the material as the testing temperature changed. This relationship is visually integrated in figure 27 for distinct comparison. Note that the *DBTT* from the geometrical parameters is coincident with that of the Charpy test. At this temperature, the *DD* density also starts to

decrease drastically which, if added together, indicates that a change in the predominant crack nucleation mechanism has begun to occur. However, this change in mechanism does not proceed directly from the ductile to the cleavage mode. Instead, ductile fracture is linked to cleavage fracture through a hybrid mode that can be named the *quasicleavage mode* (*QCM*, not to be confused with *QC* facets defined before). Defining the critical cleavage stress σ_c as the stress on the cleavage plane of the most favorable oriented grain:^[24]

$$\sigma_c = \sigma_a \times \cos\beta \quad (9)$$

where:

σ_c = cleavage critical stress

σ_a = applied stress

β = angle between cleavage plane of most favorably-oriented grain and direction of σ_a .

A qualitative description of the *DBT* and of the different fracture mechanisms involved, with special emphasis on the least understood *QCM* can be proposed and explained using a simplified conceptual illustration of the stress-temperature dependence (figure 28). When testing at high temperatures (*CVN* upper shelf), the stress in the material will first reach the yield level and plastic deformation will begin to occur. Microvoids will nucleate and grow on favorable sites with further deformation. The stress will continue to raise until microcracks form by void coalescence and propagate to complete fracture. This is a purely strain-induced *ductile fracture mode* (figure 28, $T > T_D$). On the other side of the diagram, at very low temperatures (*CVN* lower shelf), the yield stress is strongly dependent on temperature and much higher than σ_c . In this condition the material will never reach the yield level and therefore no plastic deformation can occur. Microcracks will nucleate first on favorably-oriented grains when the stress reaches the critical cleavage level σ_c . As the stress continues to increase, microcracks propagate to other grains to produce a clean undeformed fracture surface. This is a purely stress-induced *cleavage fracture mode* (figure 28, $T < T_C$).

For intermediate temperatures ($T_C < T < T_D$), if the material reaches first the yield stress level ($T_T < T < T_D$), microvoids will be nucleated on the most favorable sites. As the stress continues to increase, the material strain hardens and some planes of the most favorably oriented grains, which are already deformed to some extent, will reach the critical cleavage stress level and nucleate microcleavage cracks. These cracks will eventually produce general fracture by plastic coalescence with existing microvoids. The resulting fracture surface will show a predominance of ductile dimples with a few strain-induced cleavage^{*} facets among them. This mode is defined as *quasicleavage fracture mode type I* (QC_I). On the other hand, for the lower region of the intermediate temperatures ($T_C < T < T_T$), the critical cleavage stress is reached before yielding and therefore the most favorably-oriented grains nucleate microcleavage cracks on favored planes. These microcracks extend to the boundaries with the less favorably-oriented grains where they can not propagate because the stress is not high enough. As the stress increases and passes the yield level, those less favorably oriented grains that remained uncracked will nucleate microvoids by deformation and coalesce with the cleaved planes to produce general fracture. In this case, the fracture surface will be primarily stress-induced cleavage facets with few ductile dimples clustered in small areas among them. This mode is defined as *quasicleavage fracture mode type II* (QC_{II}).

Both types of quasicleavage fracture consist of a mixture of dimples and cleavage facets, however the amount of each one present is a function of temperature as seen in figure 24. It is also important to remember that while dimples of both quasicleavage types have the same origin (i.e., strain-induced microvoid nucleation, growth and coalescence), the cleavage facets instead are strain-induced for *type I* and stress-induced for *type II*. This different nature of the cleavage in the quasicleavage mode is suspected to be responsible for the change of the V_{QC} with temperature shown in figure 21. By similar argument then, the temperature independence of V_{DD} can be explained.

^{*}Strain-induced cleavage is a term that describes cleavage produced by a local stress-enhancement that results from strain hardening.

4.2. 3D CHARACTERIZATION OF FRACTURE

4.2.1. Shape:

The proportionality factor, a combined 3D parameter, gives expected but nonetheless important information about the characteristics of ductile dimple fracture. As seen in figure 21, V_{DD} is independent of temperature. For this material under the given conditions, V_{DD} was found to randomly fluctuate around a constant value of 0.253. This result indicates that the configuration of DD must only be a function of the shape and distribution of their nuclei. It is also important to note, however, that temperature, as seen in figure 24, has a very strong influence on the number of DD that are nucleated during the process of fracture.

On the other hand, V_{QC} shows a smooth *DBT* curve (figure 21) with upper and lower shelves just below V_{DD} and above zero respectively. These two limiting values of V_{QC} are significant in fracture characterization since they separately provide information about the fracture mechanism correlation and about the material microstructure. With the upper limit almost tangent to the constant value of V_{DD} , it is reconfirmed that at high temperature, both DD and QC are strain-induced fracture modes with the same origins.^[3,24,25] The lower limit instead has a value of approximately .08 for this material, which must be a function of the average grain size and the preferred cleavage plane orientation. This limit value will also be fixed for a given material and set of initial thermomechanical conditions. There could be, however, more than one equally preferred cleavage plane, or different planes could be preferred at different temperatures which in addition to changing the lower V_{QC} limit may also be an important factor in determining the width and shape of the temperature transition region. Further research combining 3D fractographic analysis with metallurgical analysis is suggested to investigate this topic.

4.2.2. Roughness:

The roughness factor F compiles all the 3D quantitative information measured from the specimen. As seen in figure 25, F follows closely the DBT exhibited by the material which is a consequence of its relationship to the total surface area created during the fracture process. The use of this factor F could then be extended as a method to estimate the energy required for fracture, becoming of great usefulness in failure analysis. More expeditious measurements of F could perhaps be attempted using a continuous-flow krypton adsorption method for ultra low surface area measurements.^[26,27] Preliminary experiments with this technique show that fracture surface area measurements on Charpy specimens require the maximum sensitivity of the instrumentation. Interpretation of these results still to be determined.

5. CONCLUSIONS

In polycrystalline materials with a homogeneous distribution of nucleation sites for microvoids, the fracture surface would appear 100% ductile dimples if the testing temperature is high enough to activate all those nucleation sites through strain induced microvoid coalescence. Lower temperatures allow fewer nucleation sites to become active and therefore cracking will occur on remaining plastically deformed grains through strain-induced cleavage, producing a mixture of quasicleavage facets and ductile dimples in the fracture surface.

The three dimensional geometrical factor that describes the shape of ductile dimples is independent of temperature and may only be a function of the shape and distribution of their nuclei. Instead the shape of quasicleavage facets is a function of temperature that follows closely the ductile to brittle transition curve of the Charpy impact energy. Their lower and upper limit are set by average values of grain size and ductile dimple size respectively.

The roughness factor, calculated as an indicator of the total fracture surface area, can be used as a reliable three dimensional estimator of the energy required to fracture. This estimator may prove to be very useful in failure analysis.

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FIGURES

Fig. 1: Standard dimensions of V-notch Charpy specimens.

Fig. 2: Instrumented Charpy test. a) Idealized curve. b) Actual display for 6Ni steel at 373 K.

Fig. 3: Stereoscopic measuring process (flow chart).

Fig. 4: Geometry of specimen tilt method for stereo SEM.

Fig. 5: SEM fractographs of Charpy specimens.

Fig. 6: SEM fractograph showing *DD* and *QC* according with given definition.

Fig. 7: Idealized fracture profile with 3D parameters as measured for *DD* and *QC*.

Fig. 8: Sample area on a Charpy specimen.

Fig. 9: Random sampling scheme.

Fig. 10: Charpy impact energy *CVN* vs. temperature.

Fig. 11: Average of $1/A$ vs. temperature.

Fig. 12: Histogram of the projected area for all features.

Fig. 13: Histogram of the projected area for *DD*.

Fig. 14: Histogram of the projected area for *QC*.

Fig. 15: Average of maximum depth vs. temperature.

Fig. 16: Histograms of the maximum depth for all features.

Fig. 17: Histograms of the maximum depth for *DD*.

Fig. 18: Histograms of the maximum depth for *QC*.

Fig. 19: Average proportionality factor vs. temperature.

Fig. 20: Histograms of the proportionality factor for all features.

Fig. 21: Average proportionality factor for *DD* and *QC* vs. temperature.

Fig. 22: Histograms of the proportionality factor for *DD*.

Fig. 23: Histograms of the proportionality factor for *QC*.

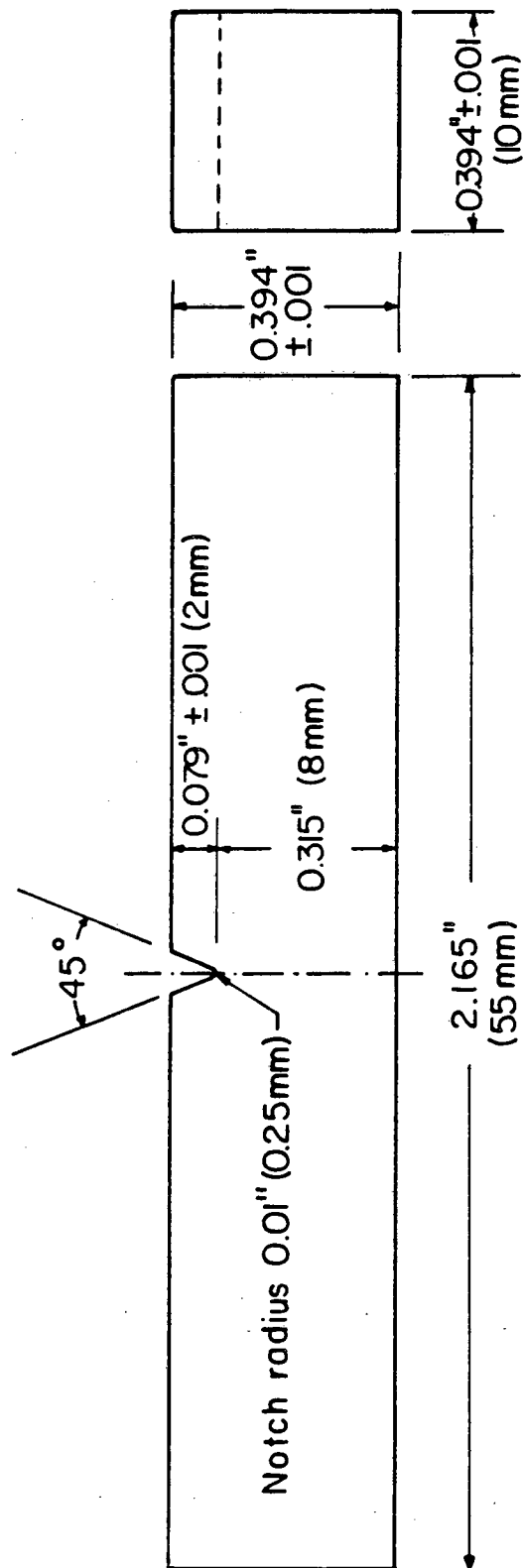
Fig. 24: Ductile dimple density vs. temperature.

Fig. 25: Roughness factor vs. temperature.

Fig. 26: Confidence levels for mean values of A , Z and V .

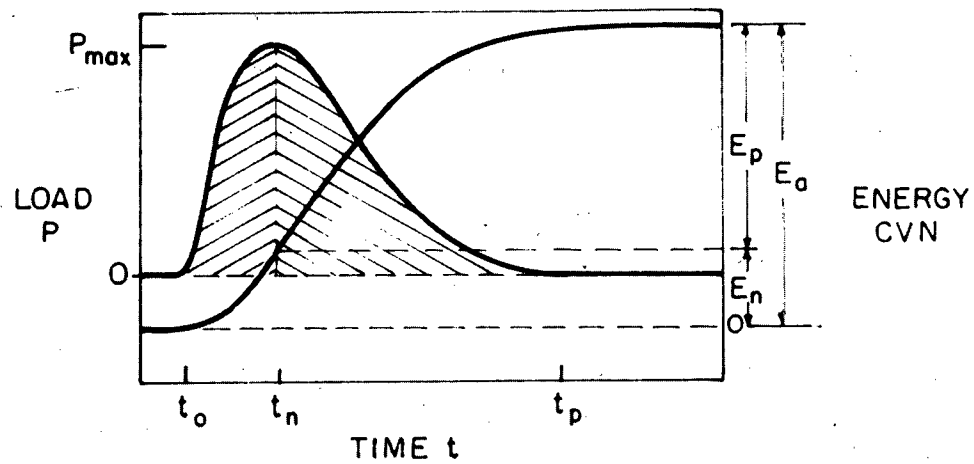
Fig. 27: Comparative plot of E_a , V_{DD-QC} , ρ and F vs. temperature.

Fig. 28: Qualitative stress-temperature dependence and fracture modes predominance.



XBL 7910-7195

Figure 1



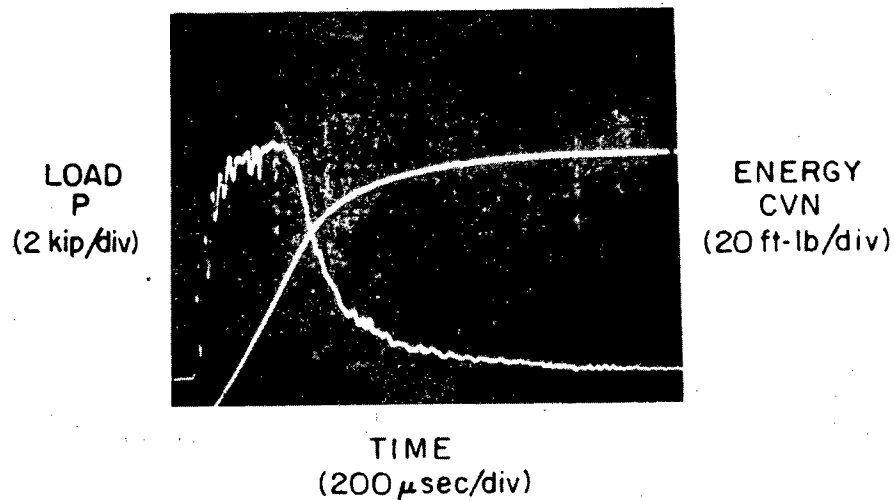
$$E_0 = E_n + E_p$$

E_0 = TOTAL ABSORBED ENERGY

E_n = ESTIMATED CRACK NUCLEATION ENERGY

E_p = ESTIMATED CRACK PROPAGATION ENERGY

(a) IDEALIZED CURVES

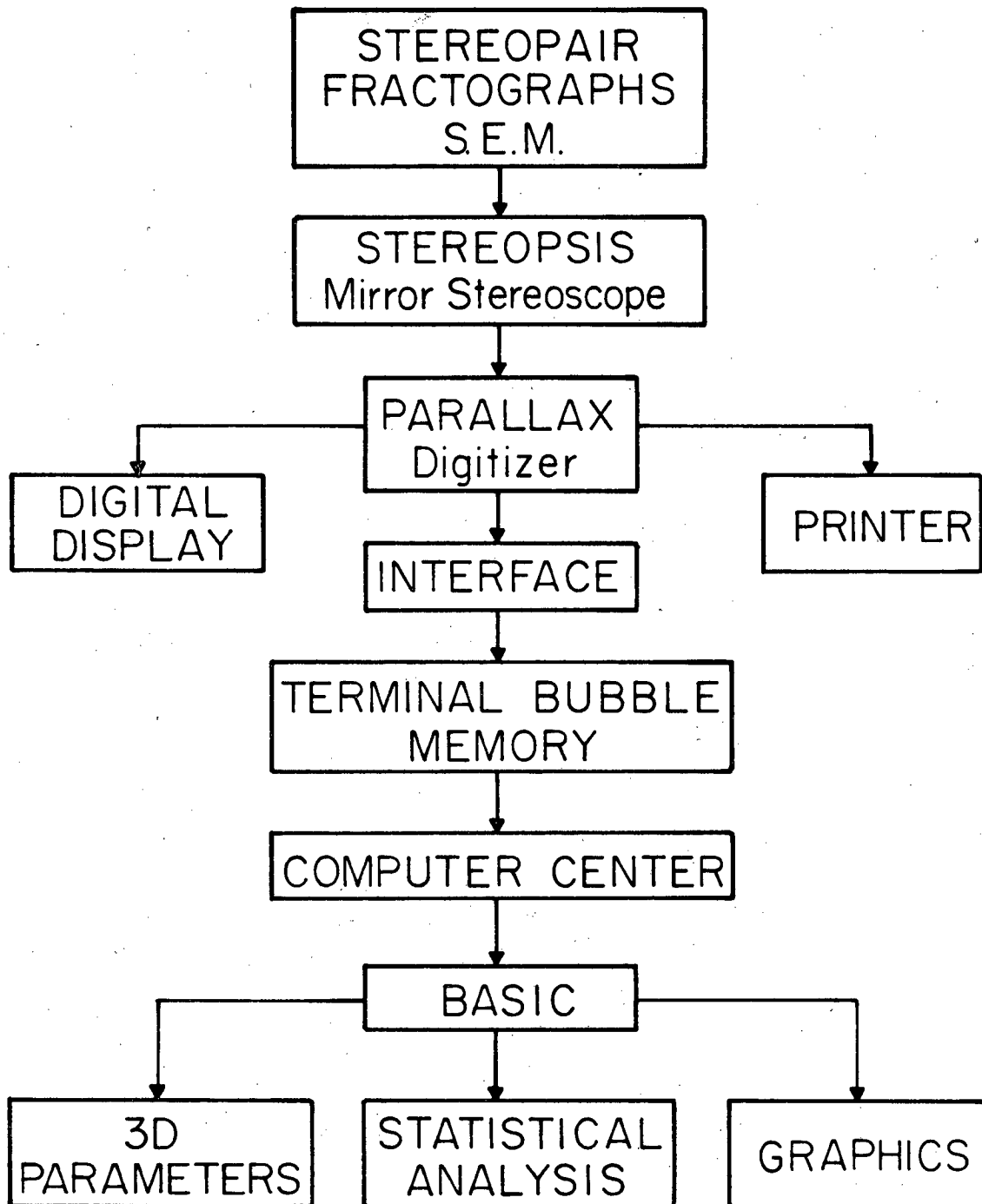


(b) ACTUAL DISPLAY FOR 6 Ni STEEL AT 373 K

INSTRUMENTED CHARPY TEST

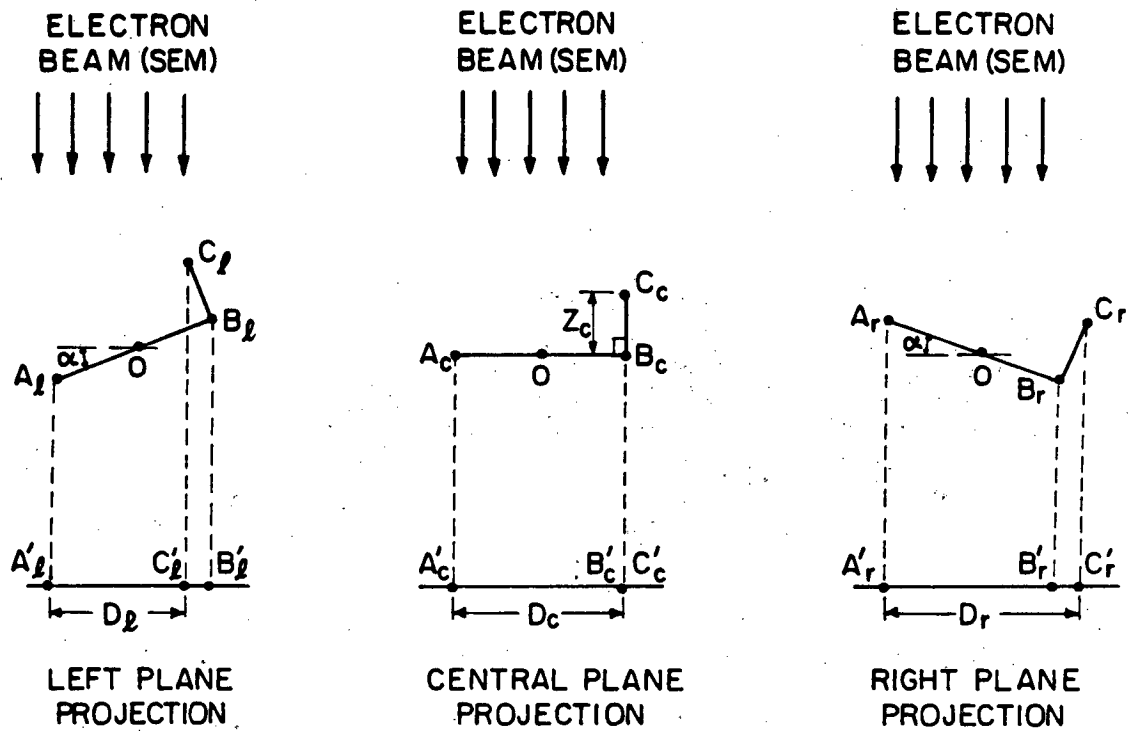
Figure 2

XBB 823-2582A



XBL 8111-12866

Figure 3



$$D_r - D_l \equiv \text{"PARALAX" } P$$

$$D_l = D_c \cos \alpha - Z_c \sin \alpha$$

$$D_r - D_l = Z_c 2 \sin \alpha$$

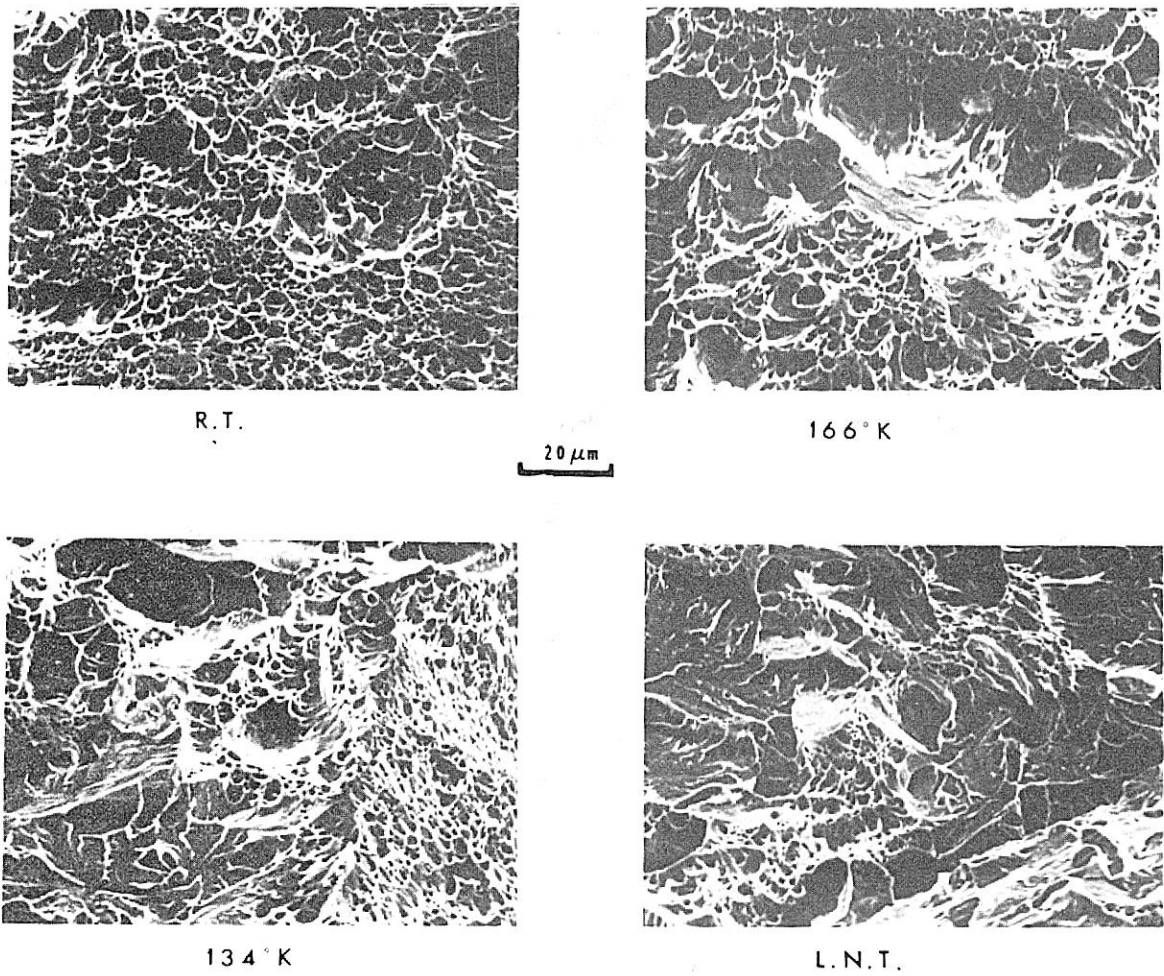
$$D_r = D_c \cos \alpha + Z_c \sin \alpha$$

$$Z_c = \frac{P}{2 \sin \alpha}$$

GEOMETRY OF SPECIMEN TILT METHOD FOR STEREO SEM
PARALLEL PROJECTION ($M > 500X$)

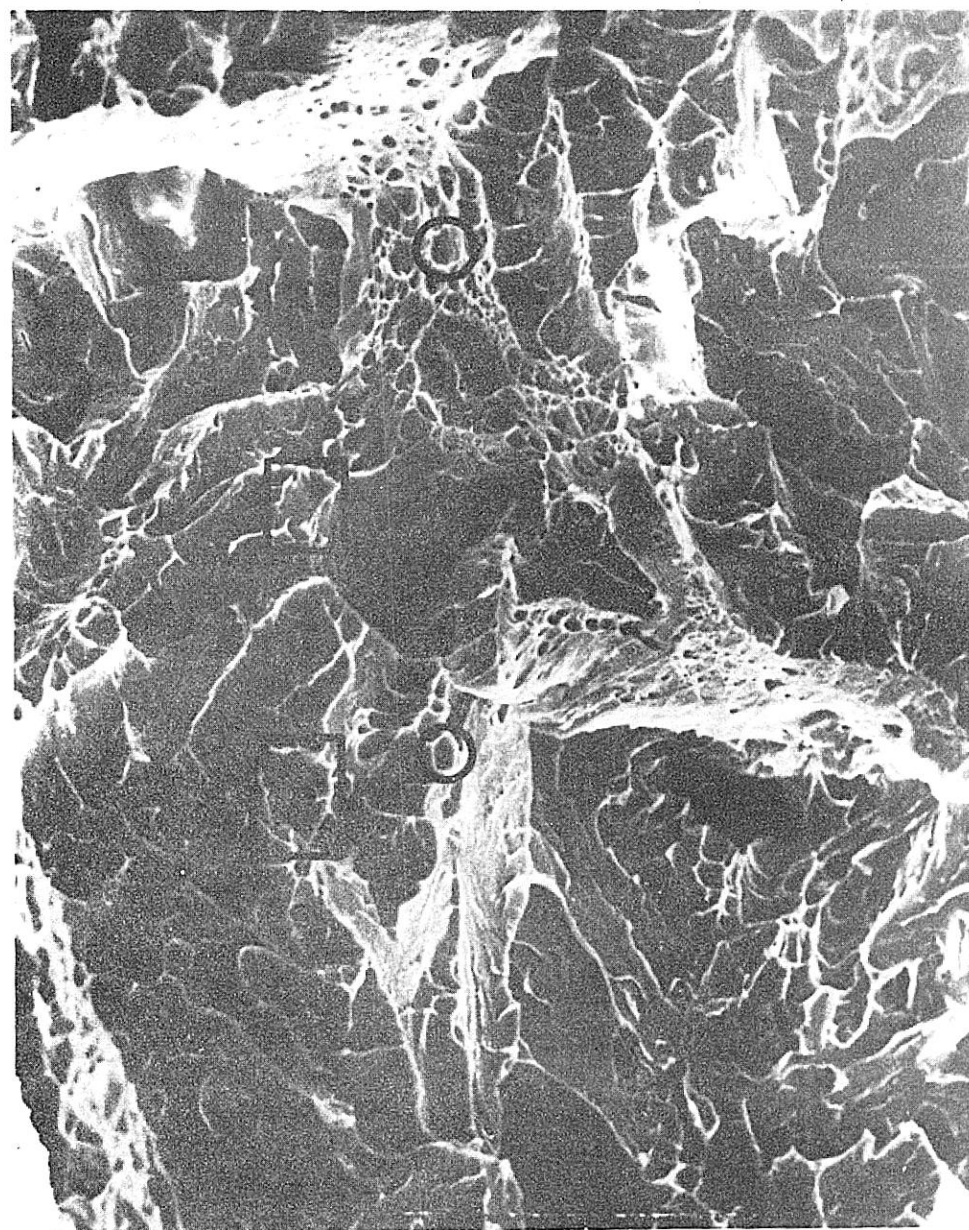
XBL 82 3-5376

Figure 4



XBB 811-67A

Figure 5



20 μm

77°K

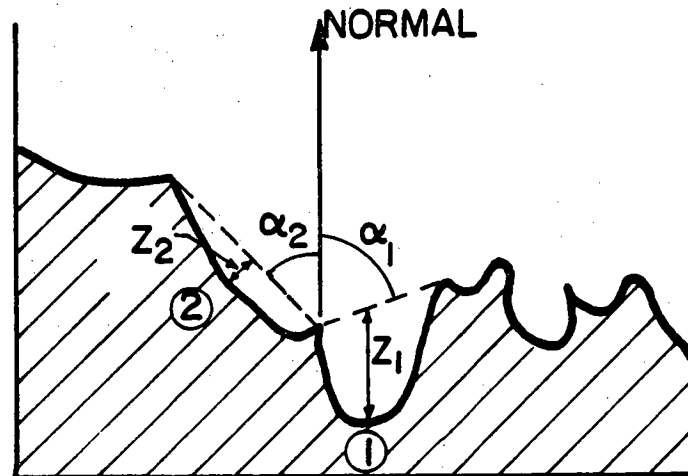
○ DUCTILE DIMPLES

□ QUASI-CLEAVAGE

Figure 6

XBB 823-1820B

FRACTURE SURFACE PROFILE



- ① DUCTILE DIMPLE (DD) PROFILE
- ② QUASI-CLEAVAGE (QC) PROFILE

3D MEASUREMENTS:

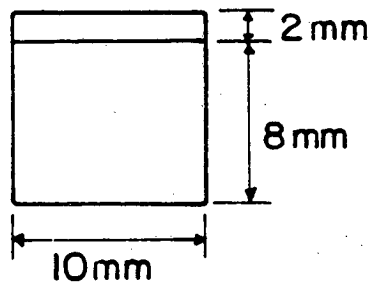
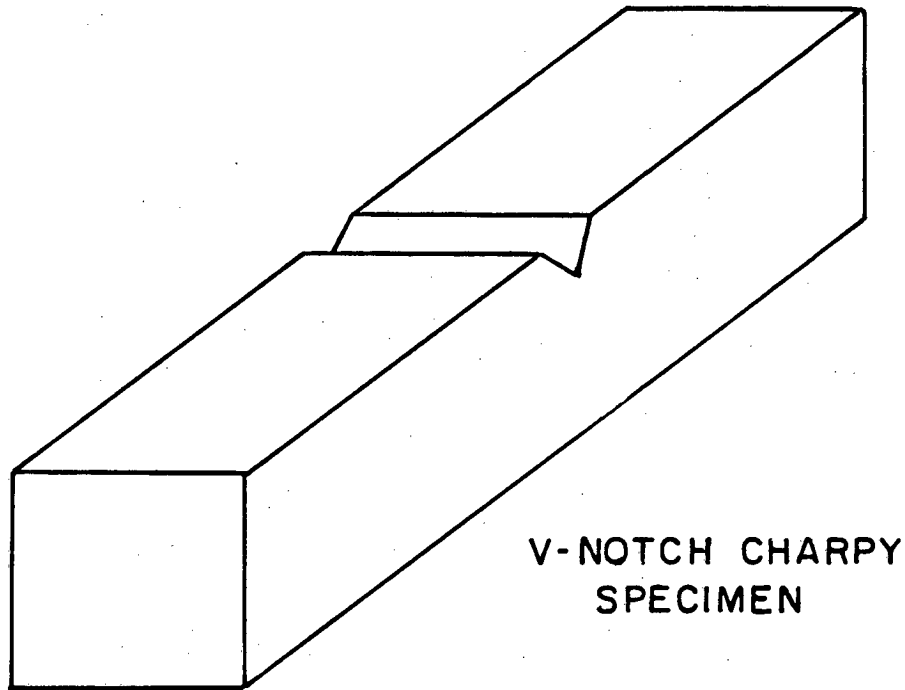
α = ANGLE OF INCLINATION or
TOPOGRAPHIC CURVATURE

Z = DEPTH (DD) or HEIGHT OF RIDGES (QC)

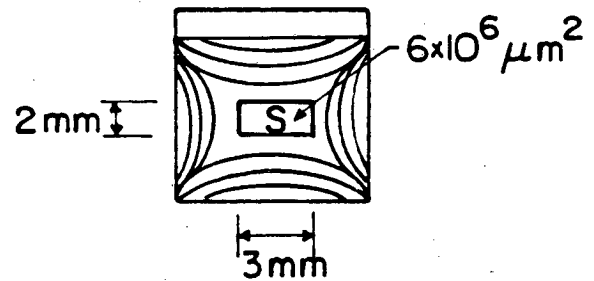
PROJECTED AREA "A" NOT SHOWN

XBL8III-6943

Figure 7



NET CROSS-SECTIONAL
AREA



TYPICAL DUCTILE FRACTURE
SURFACE

SEM FRACTOGRAPH at $2000 \times \approx 250 \mu\text{m}^2$

$S \approx 2400$ SEM FRACTOGRAPHS

Figure 8

XBL 8111-6942

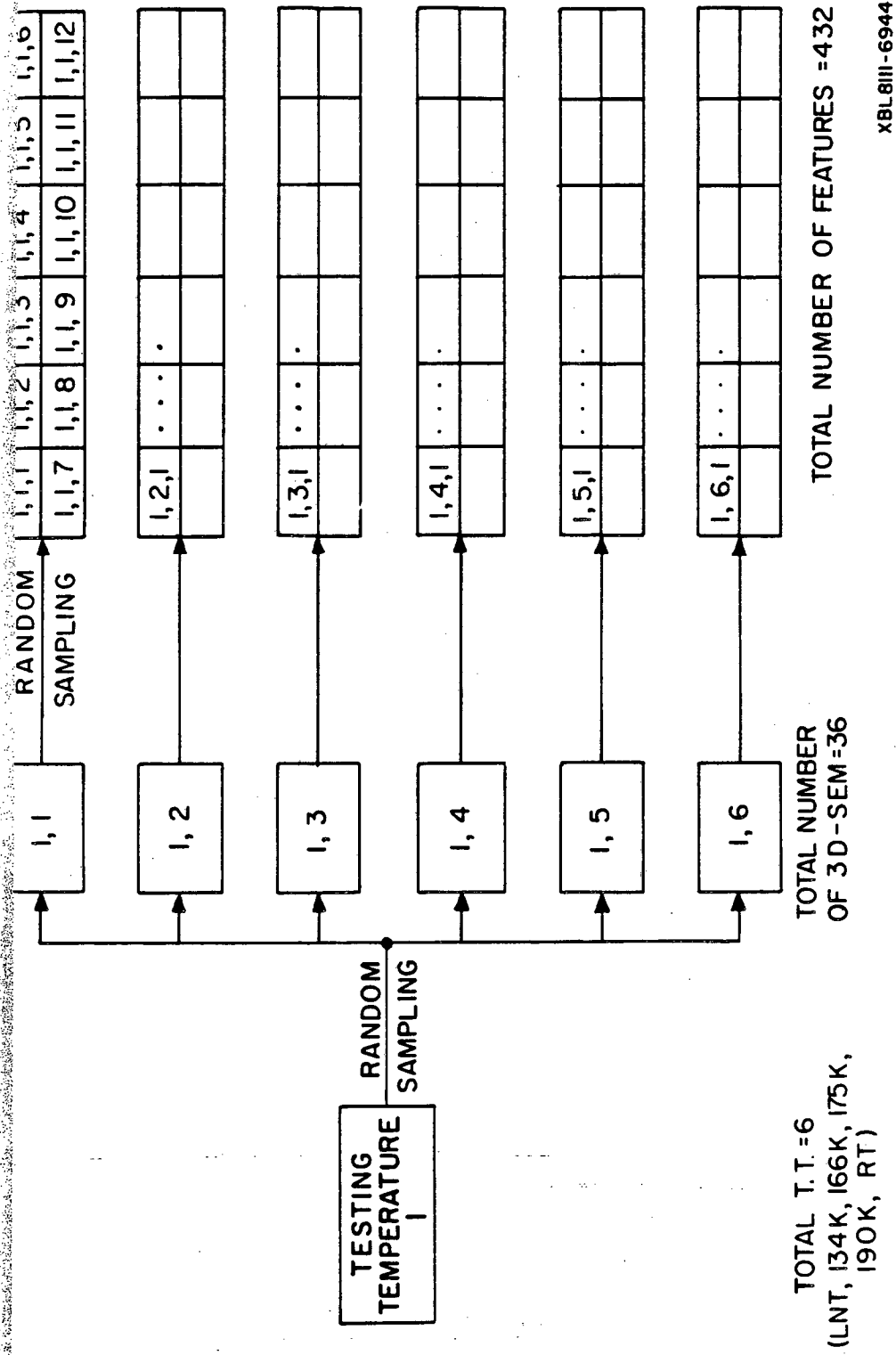
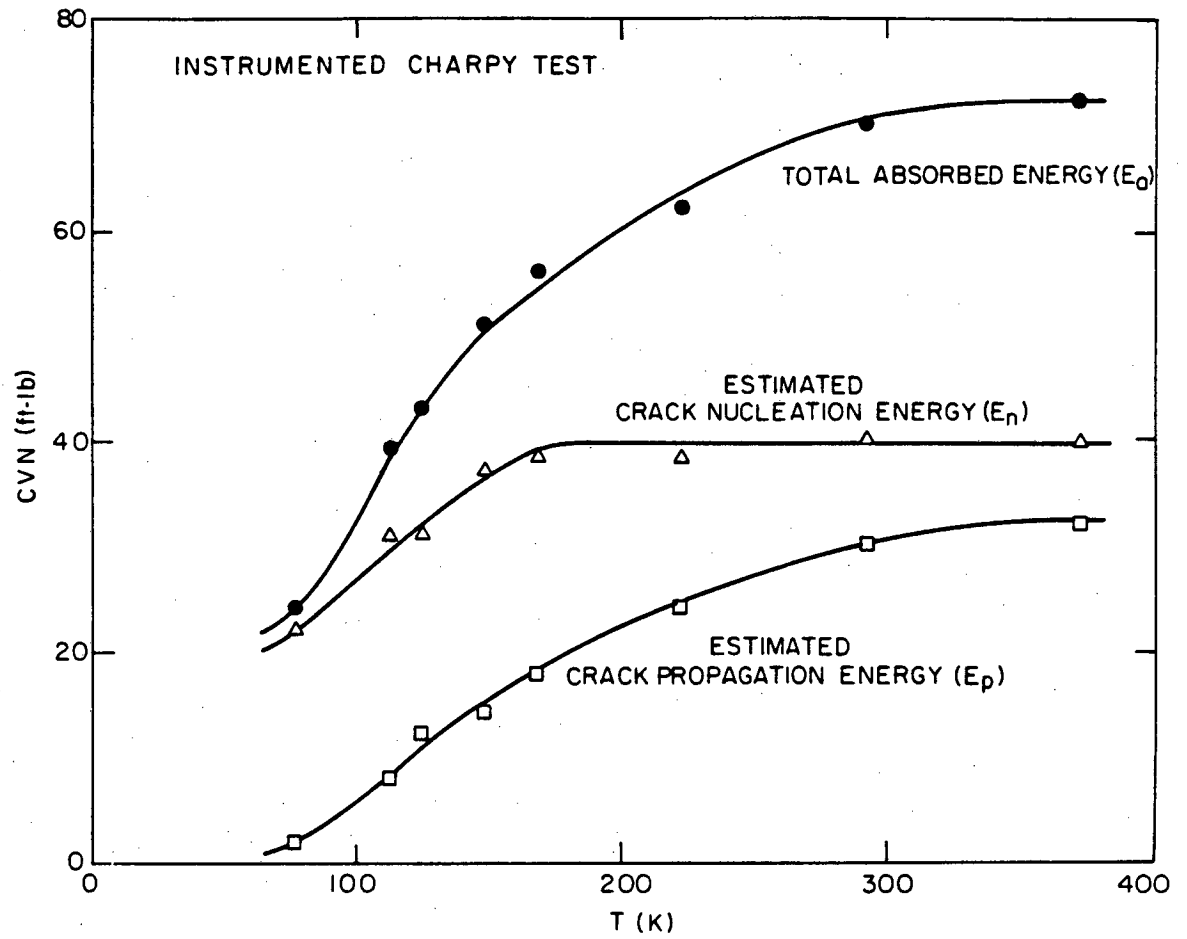
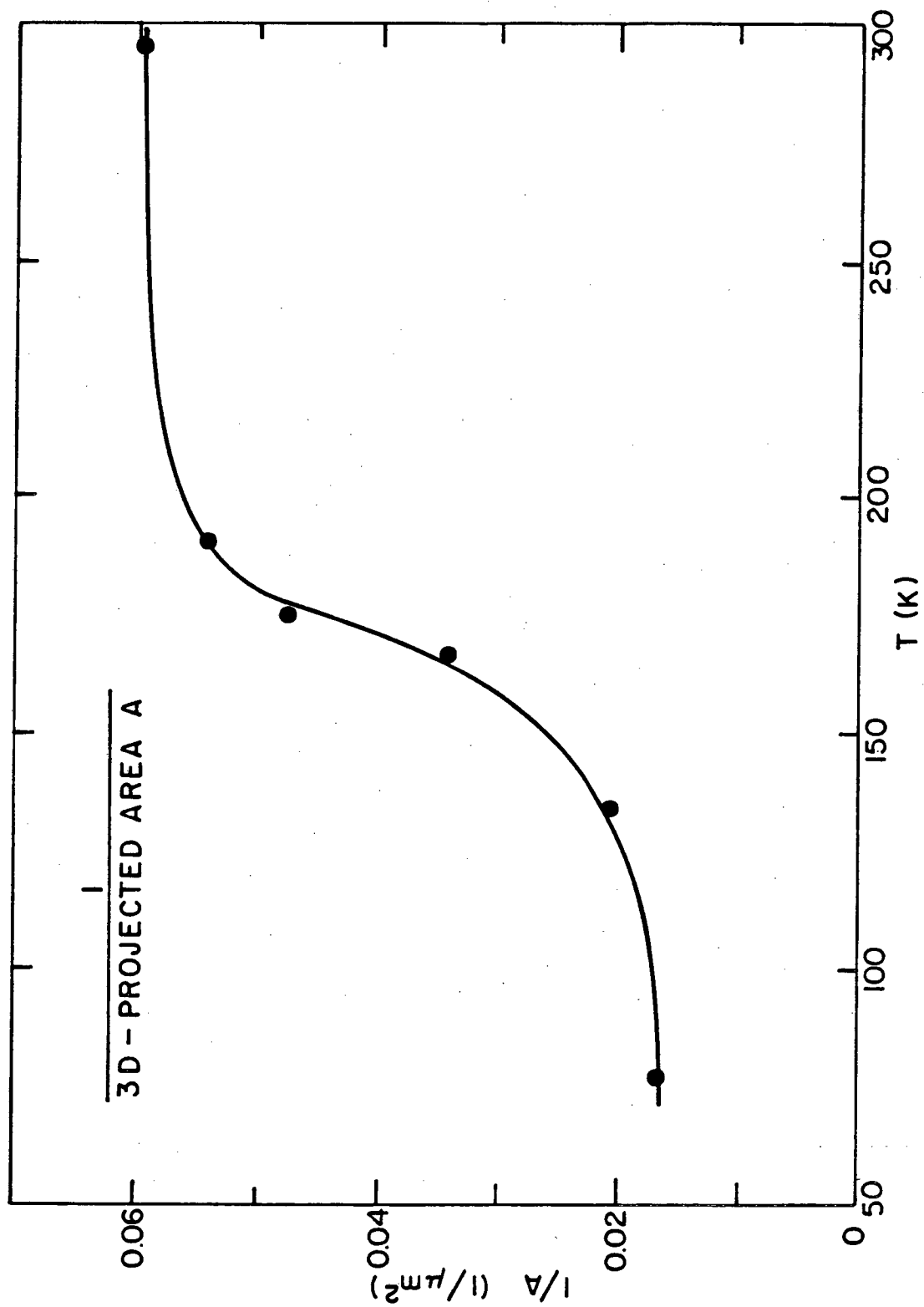


Figure 9



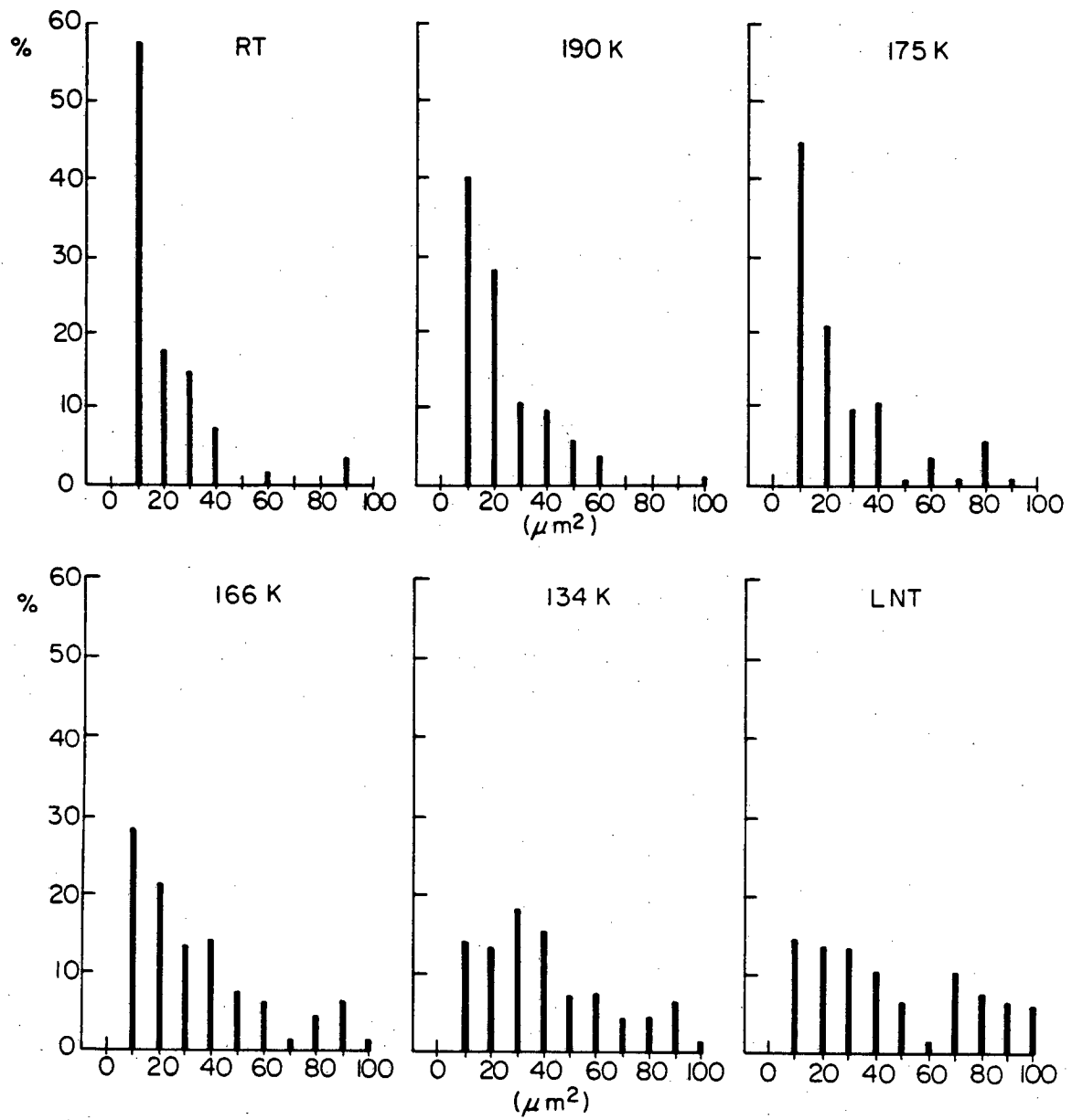
XBL823-5375

Figure 10



XBL 8110-6900

Figure 11

PROJECTED AREA, A
ALL FEATURES

XBL 821-5030

Figure 12

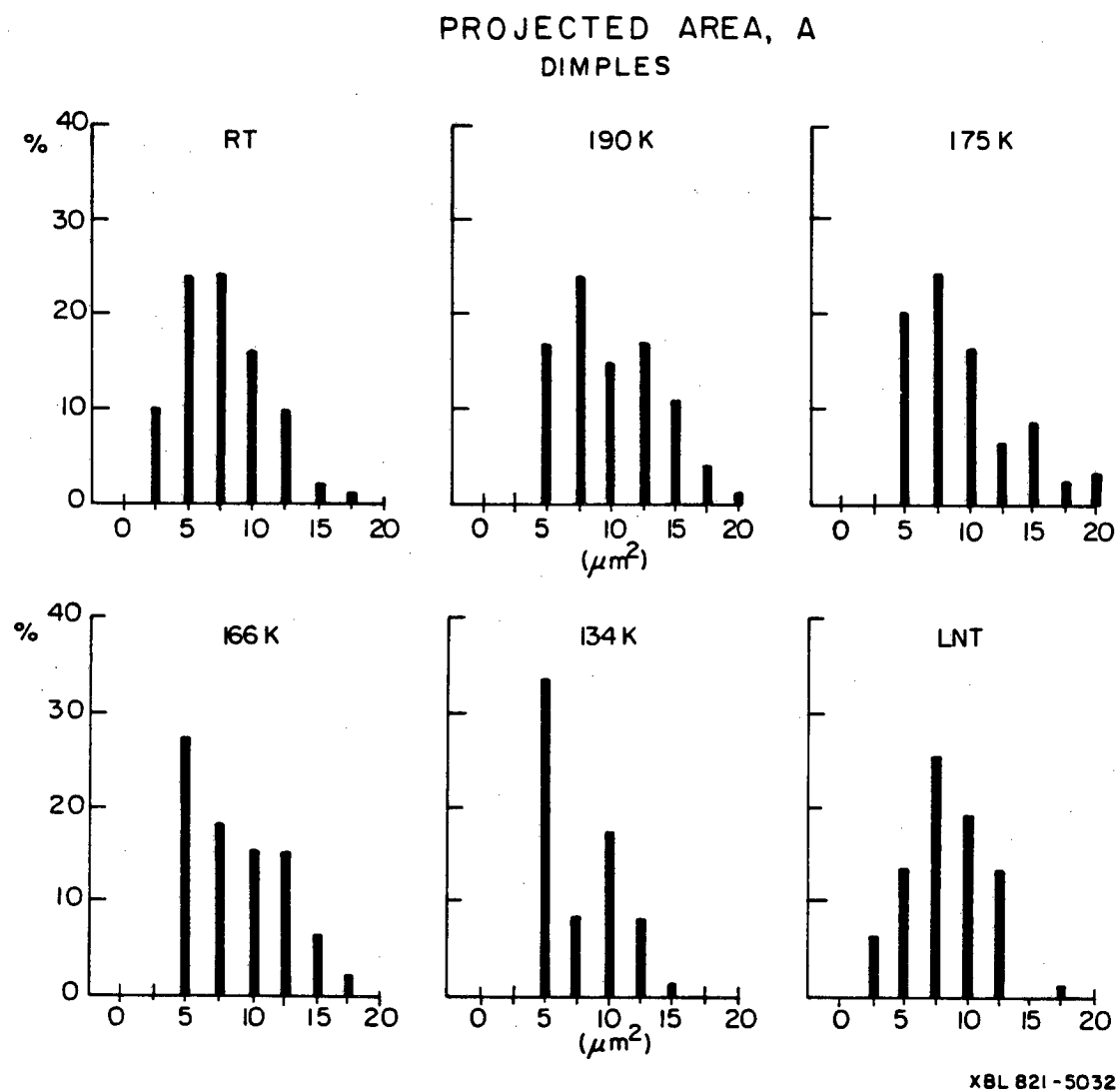
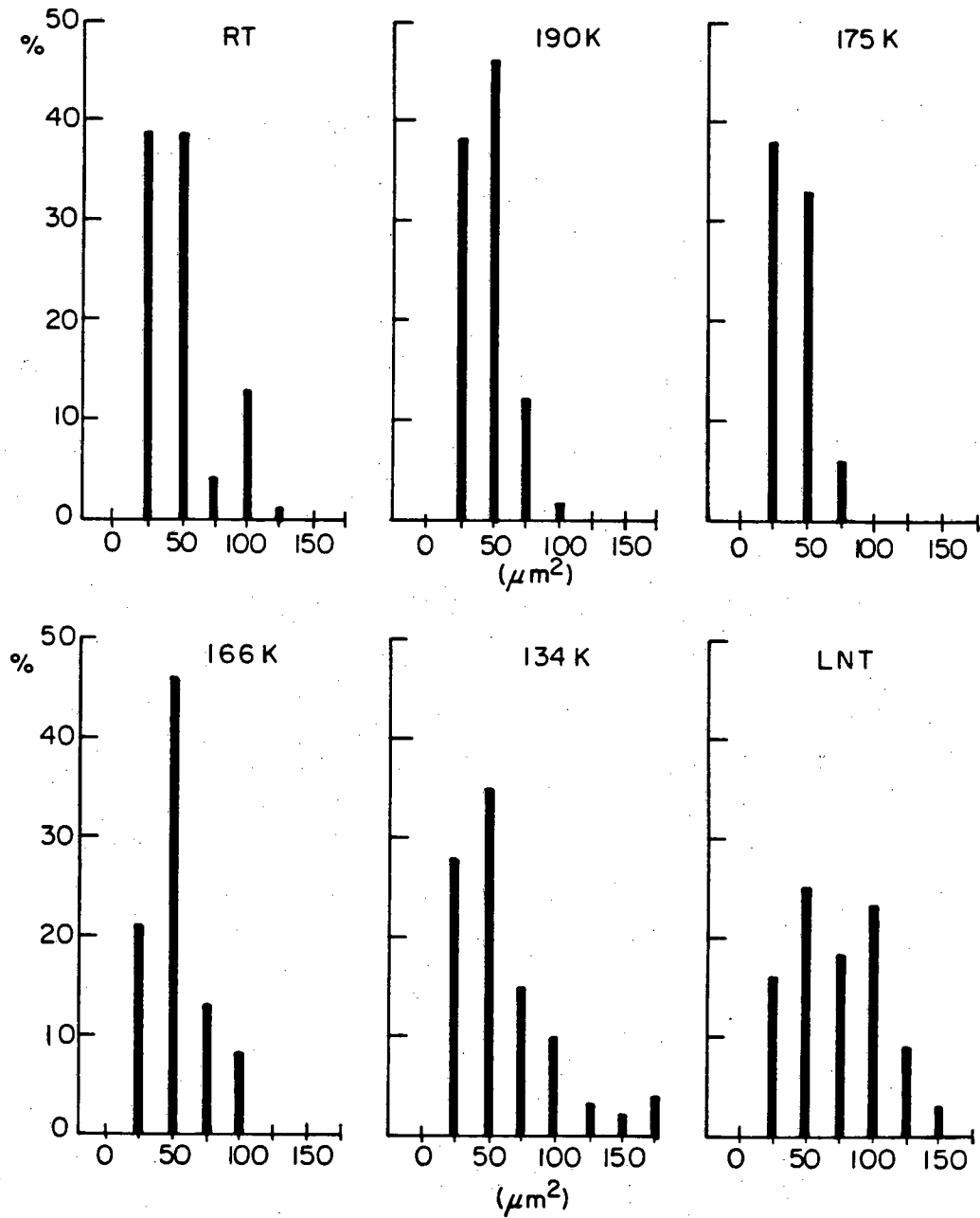


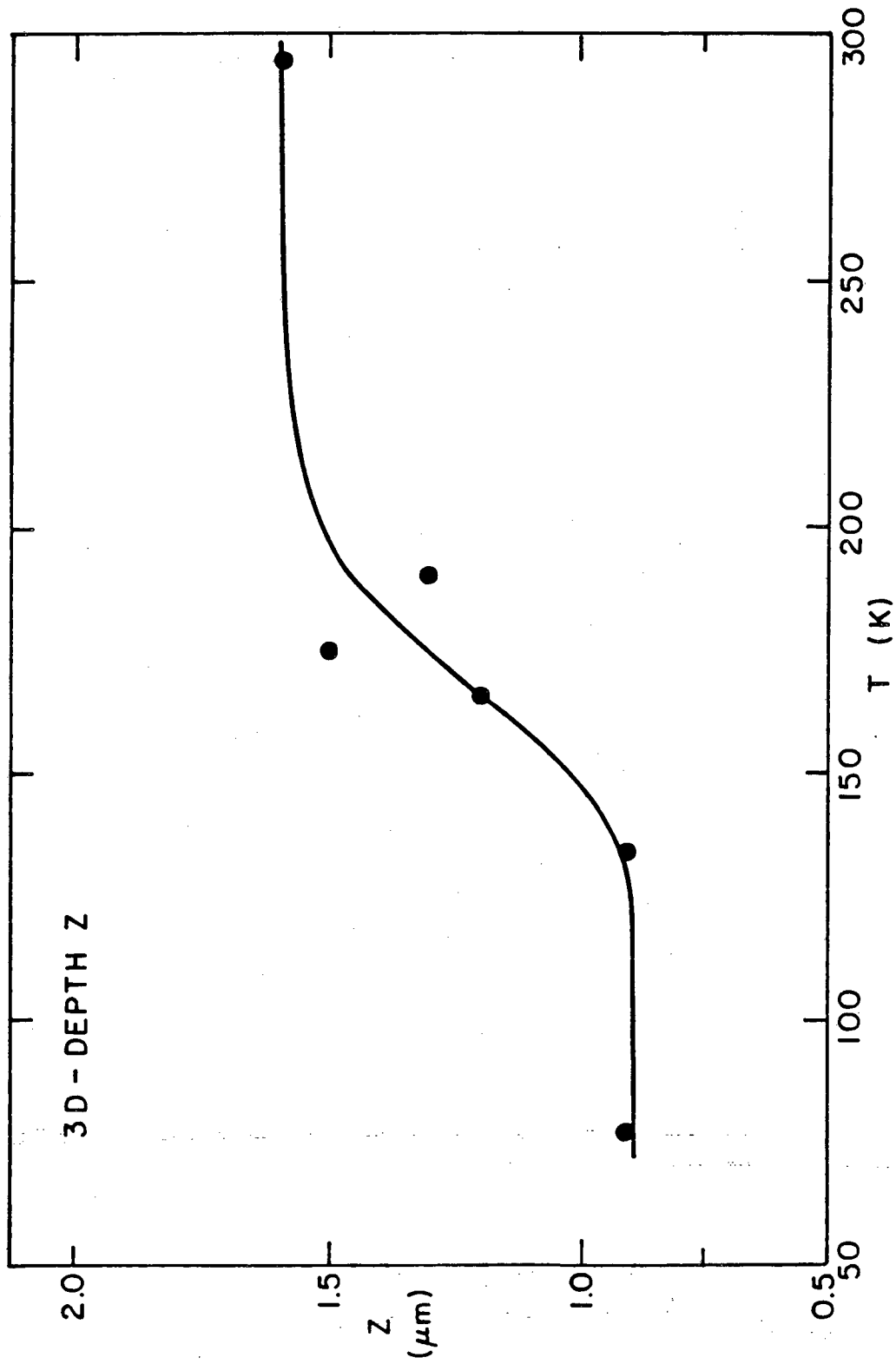
Figure 13

PROJECTED AREA, A
QUASI CLEAVAGE



XBL821-5035

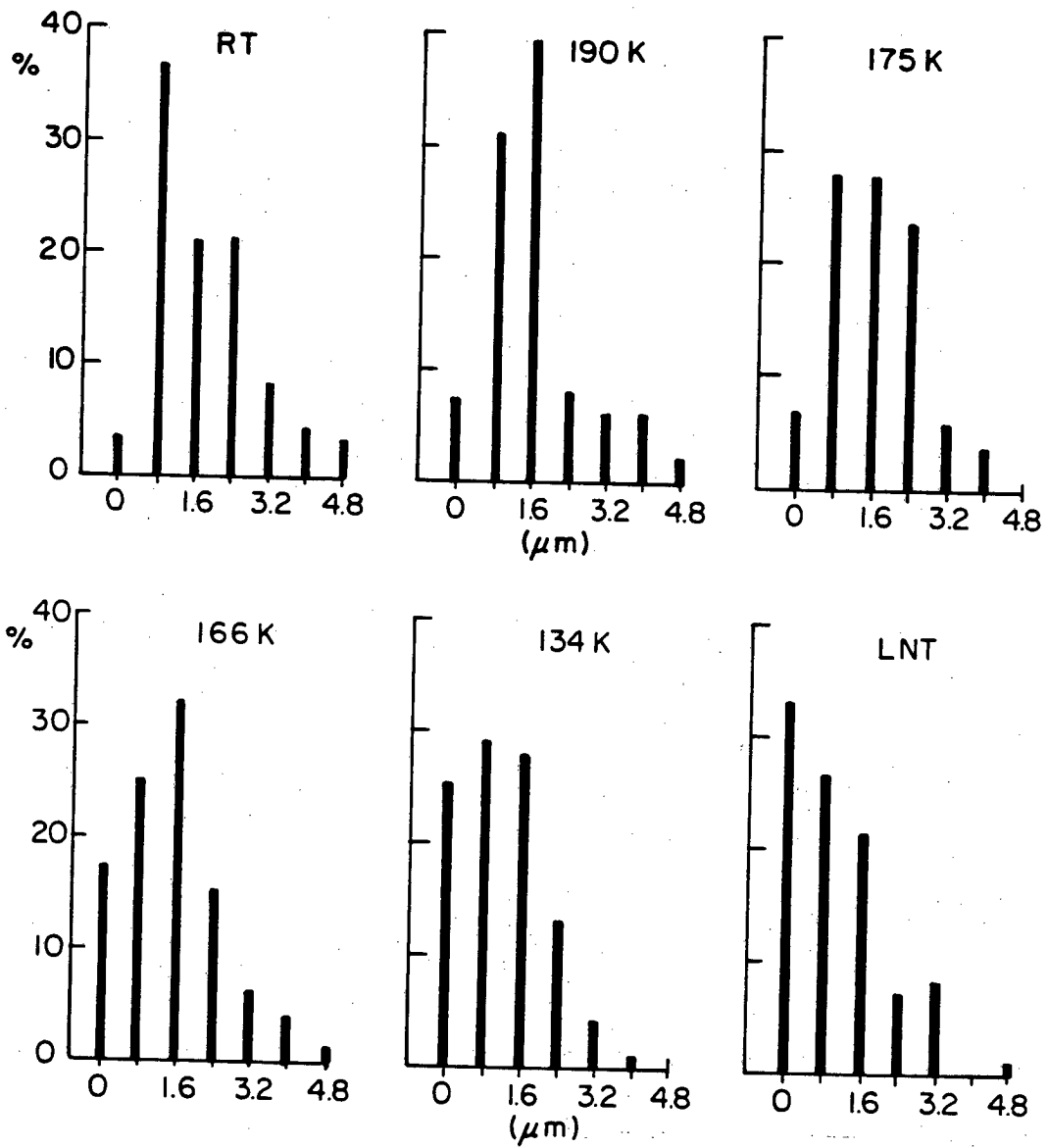
Figure 14



XBL8110-6903

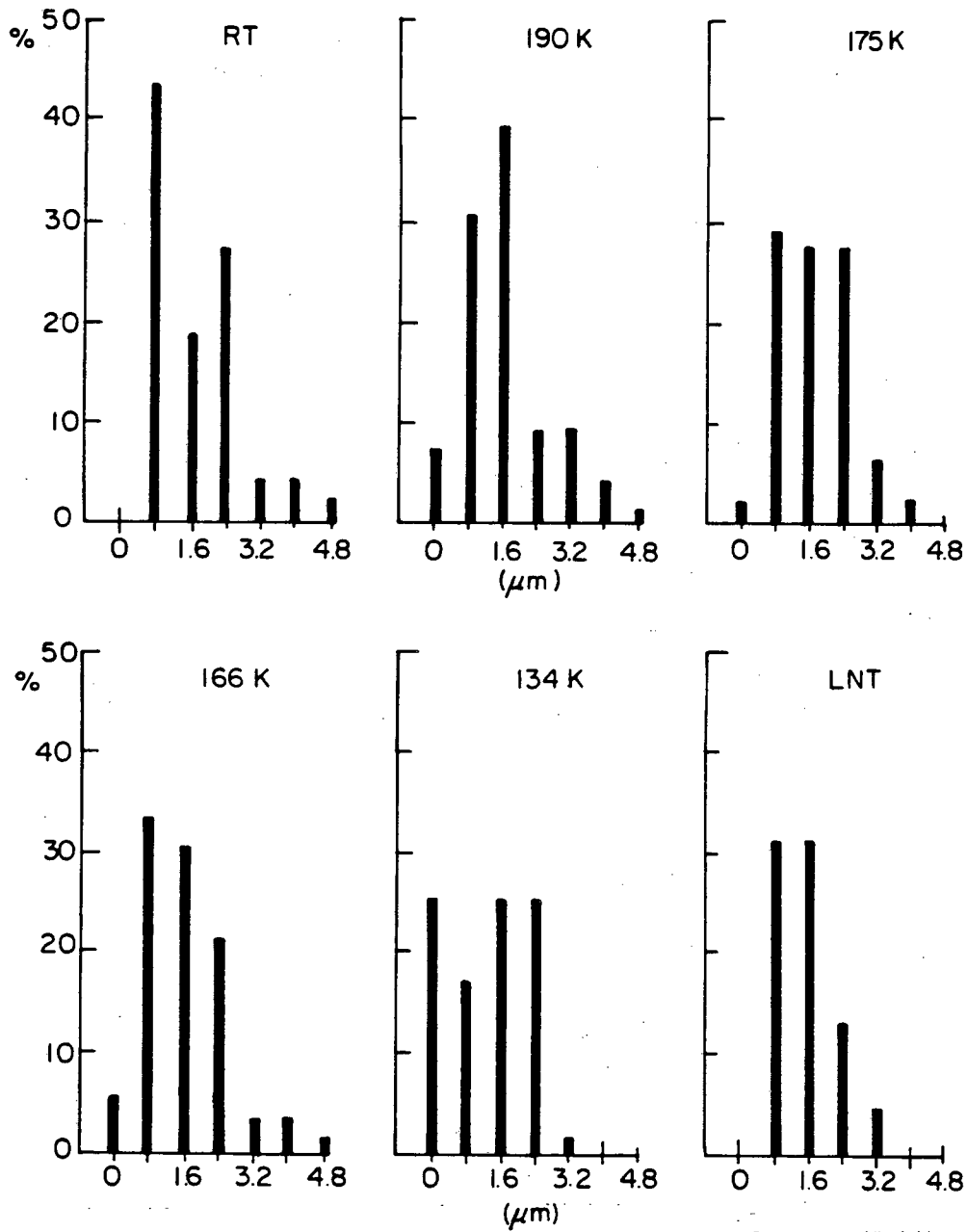
Figure 15

MAXIMUM DEPTH, Z
ALL FEATURES



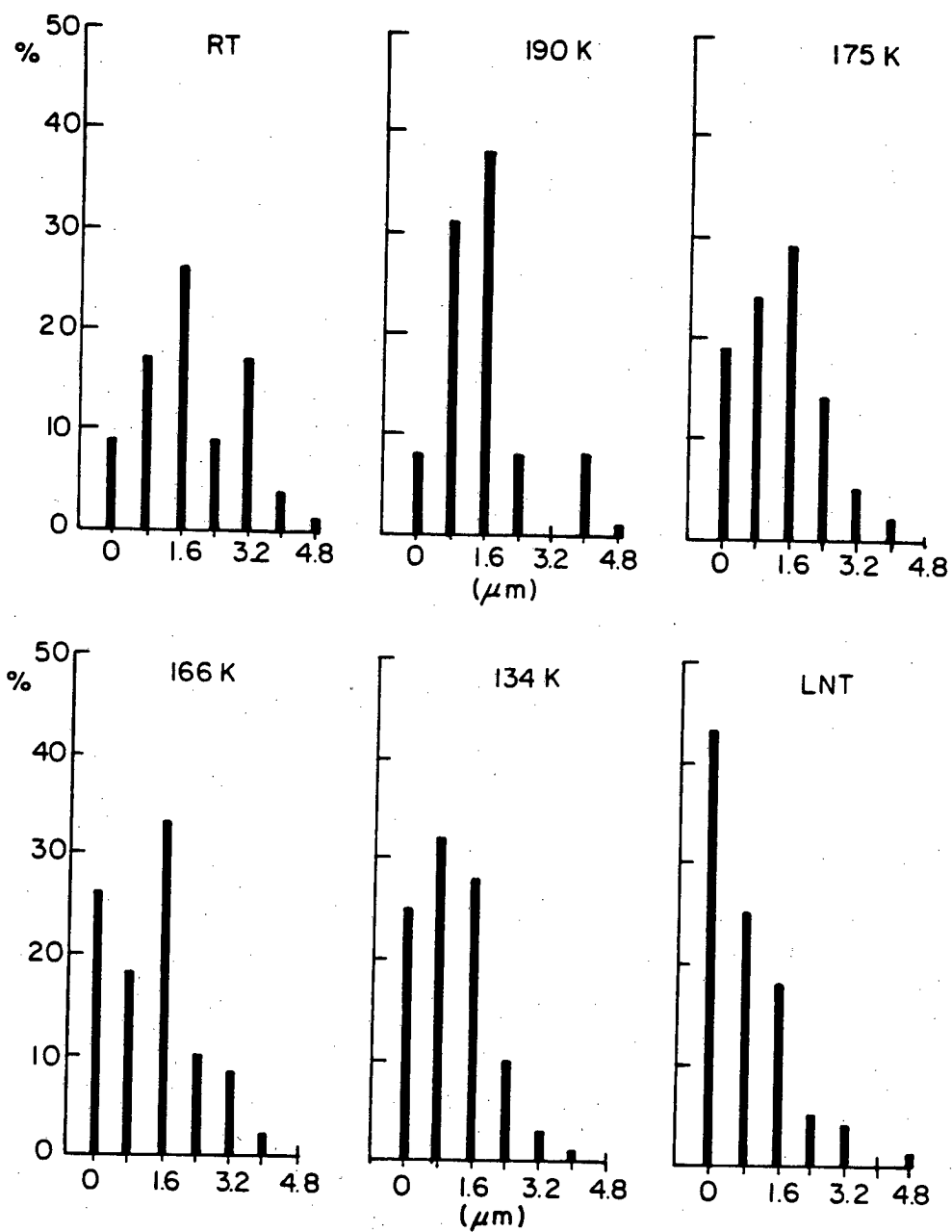
XBL82I-5029

Figure 16

MAXIMUM DEPTH, Z
DIMPLES

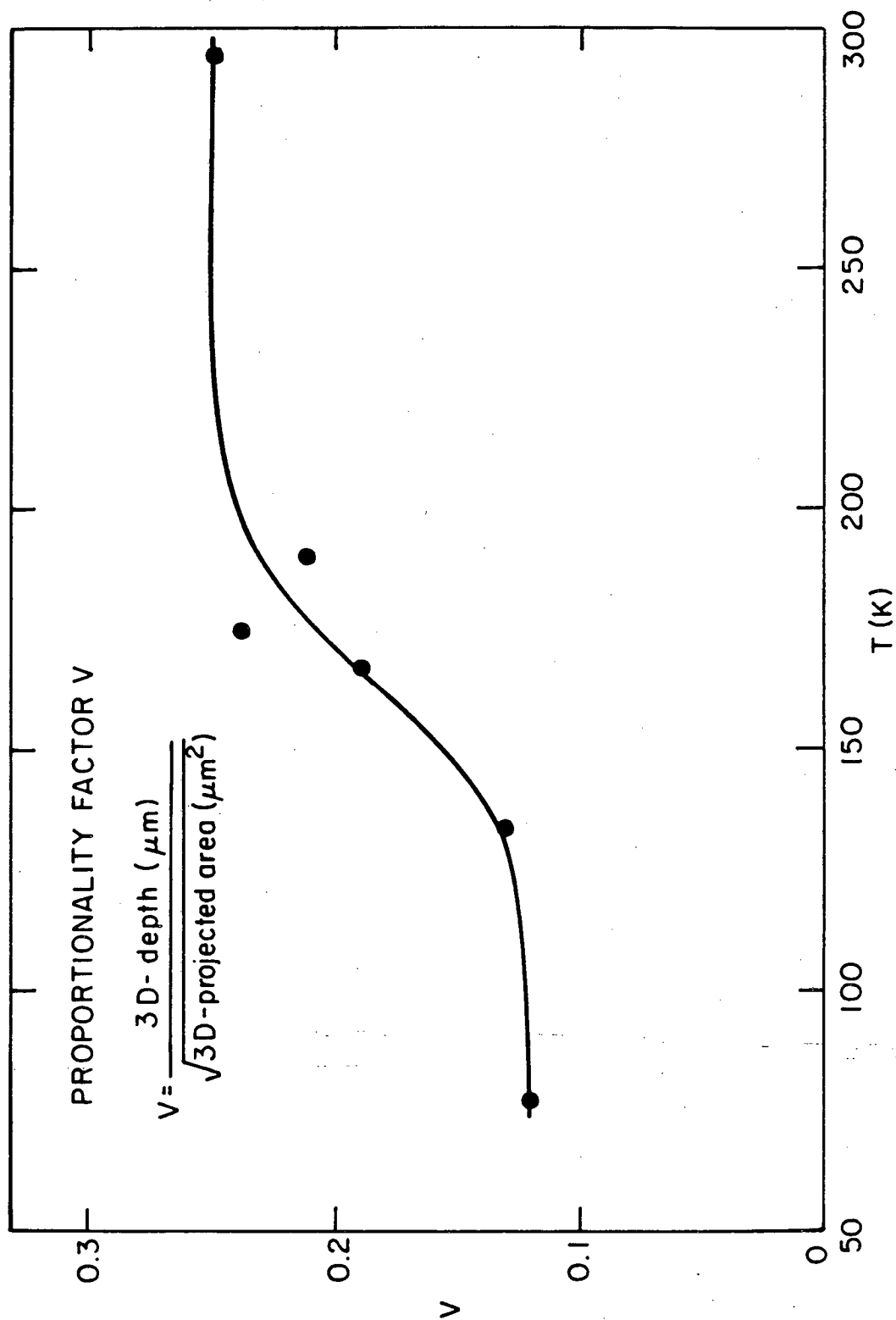
XBL 821-5031

Figure 17

MAXIMUM DEPTH, Z
QUASI CLEAVAGE

XBL 82-5034

Figure 18



XBL 823-5373

Figure 19

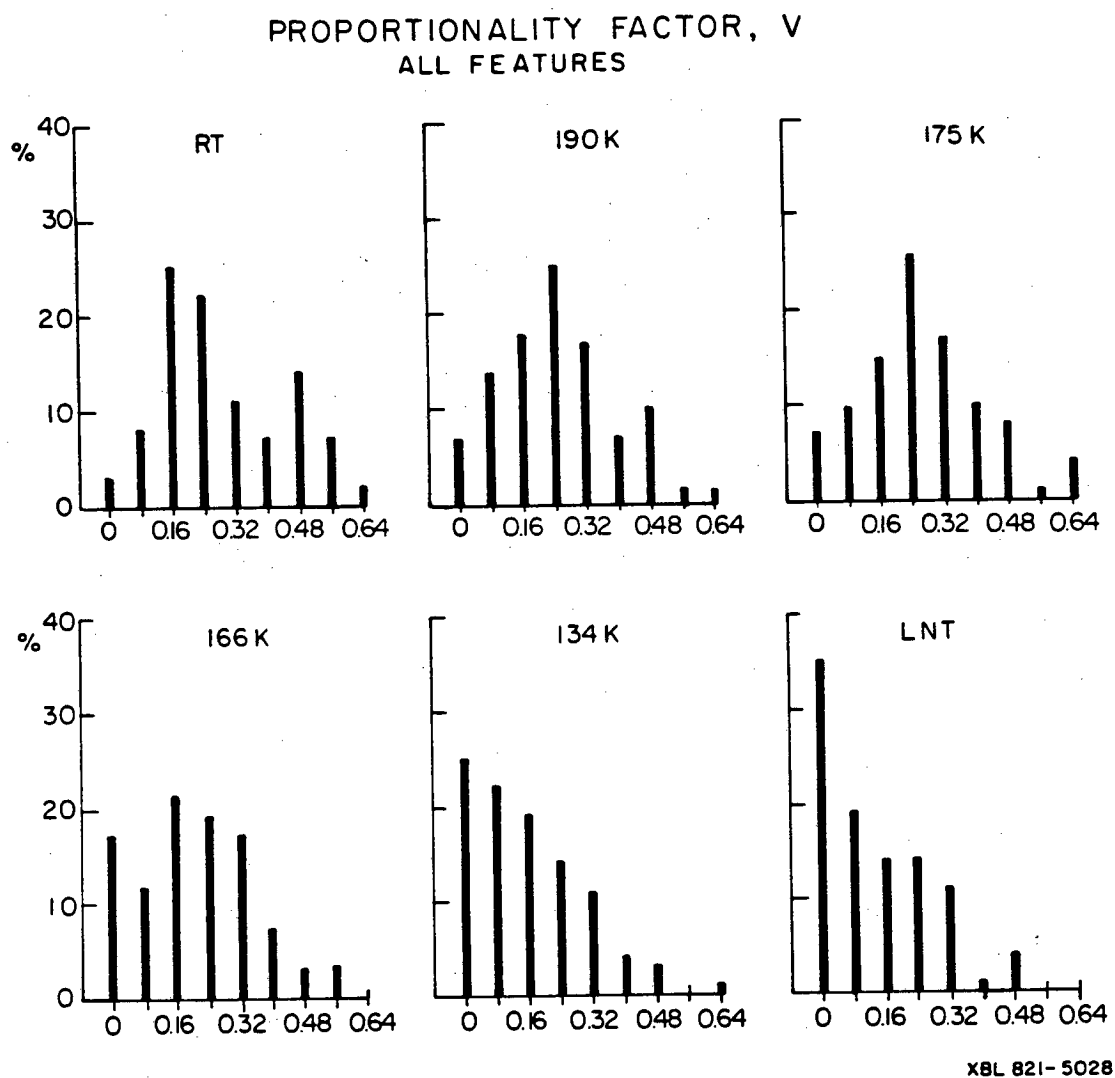
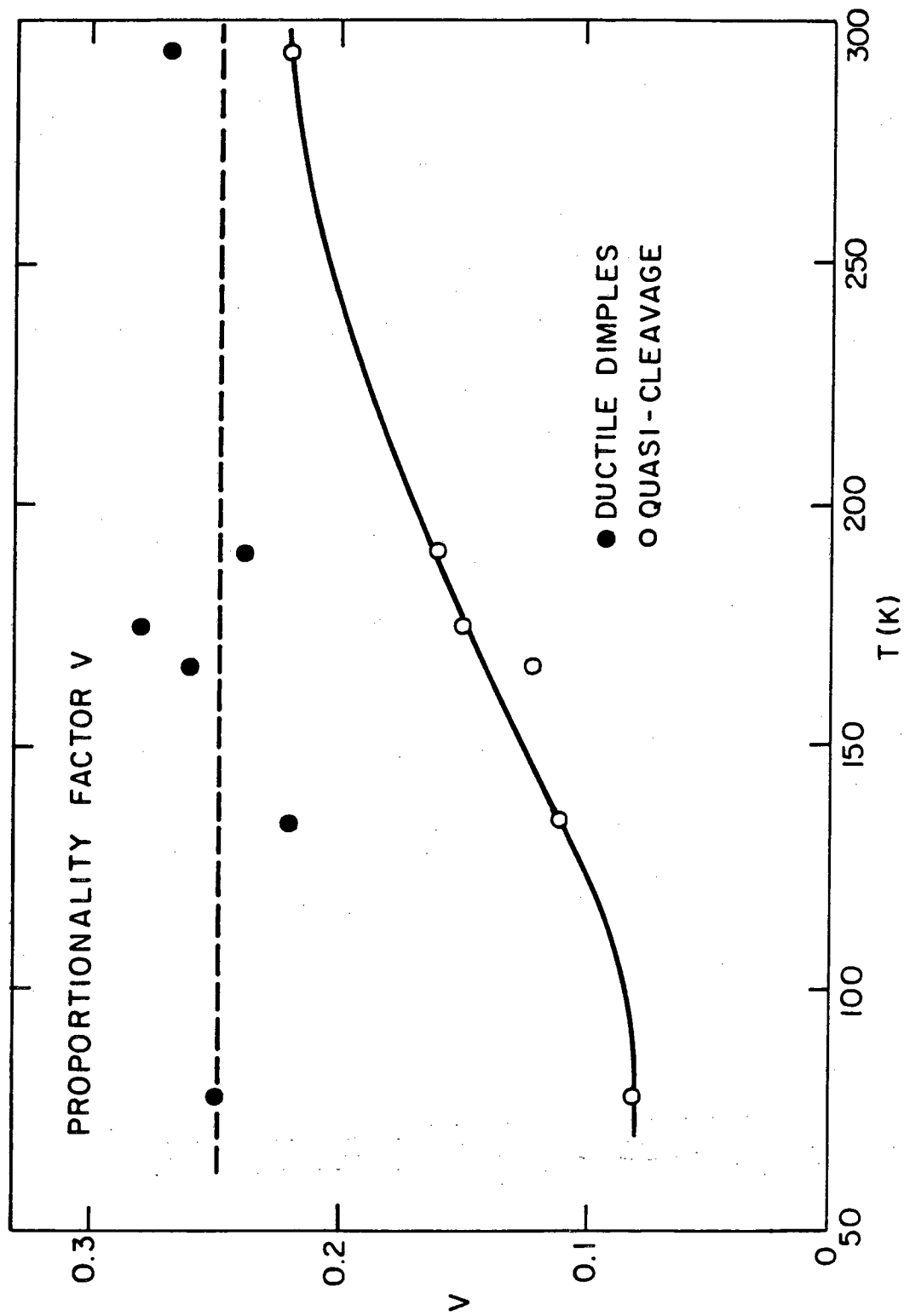
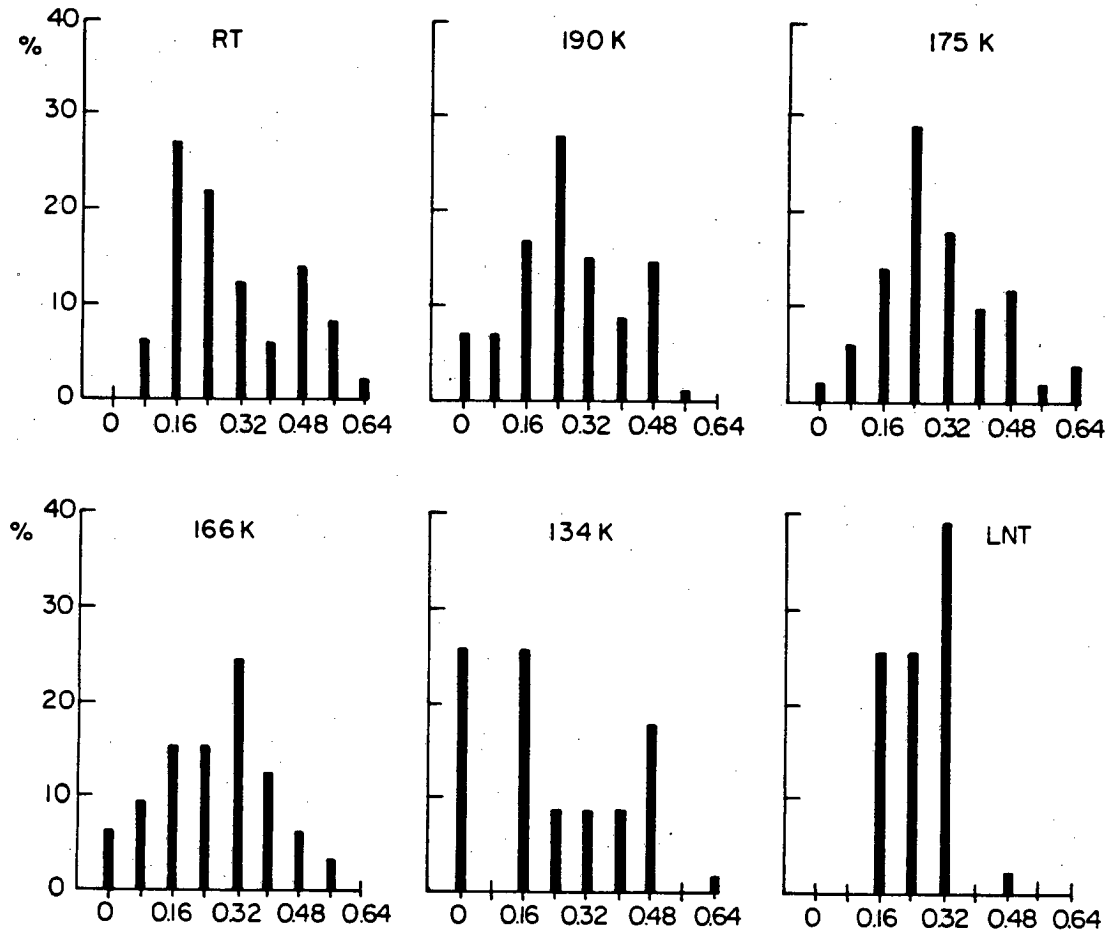


Figure 20



XBL 823-5374

Figure 21

PROPORTIONALITY FACTOR, V
DIMPLES

XBL 821-5033

Figure 22

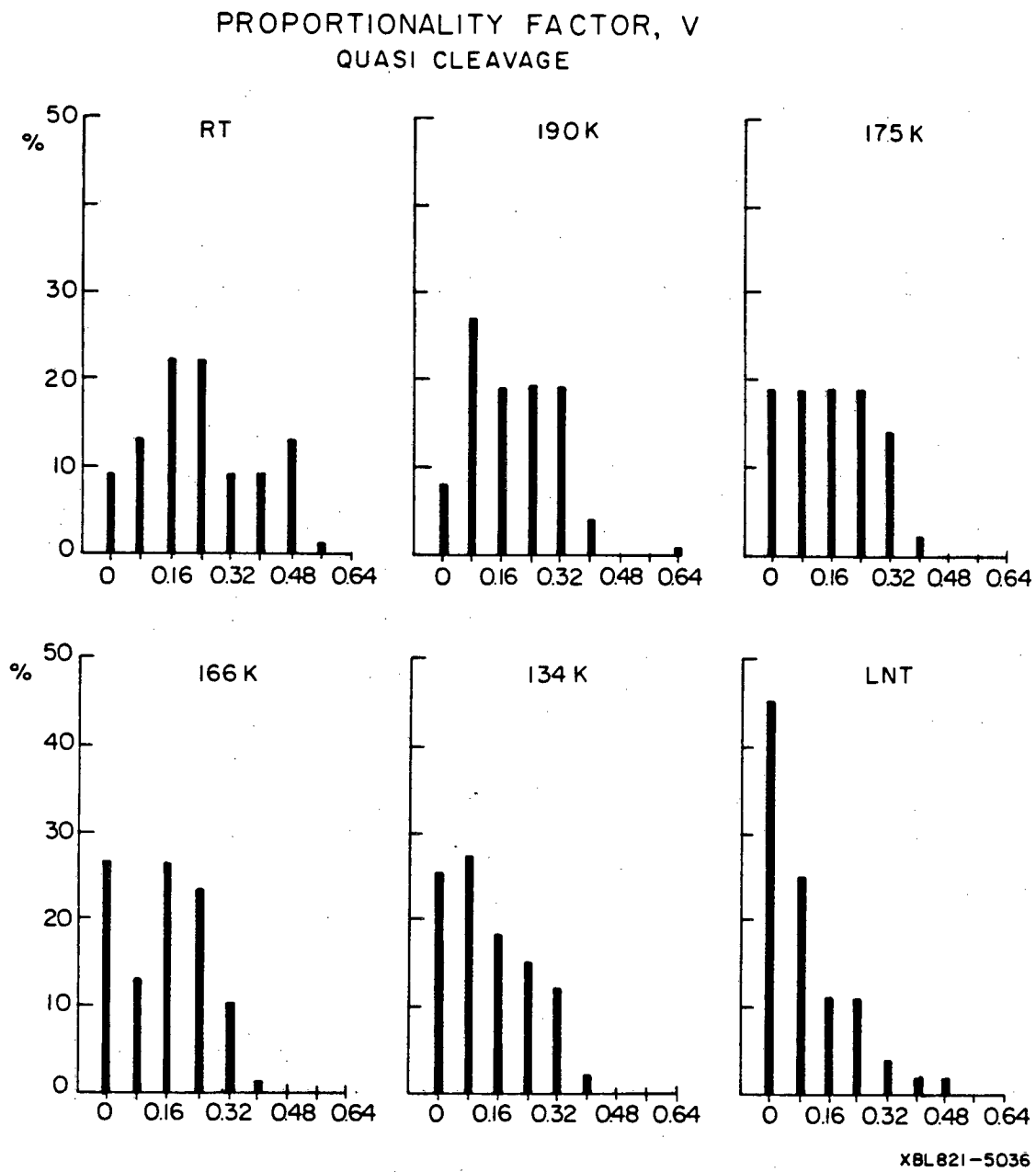


Figure 23

XBL 8110-6898

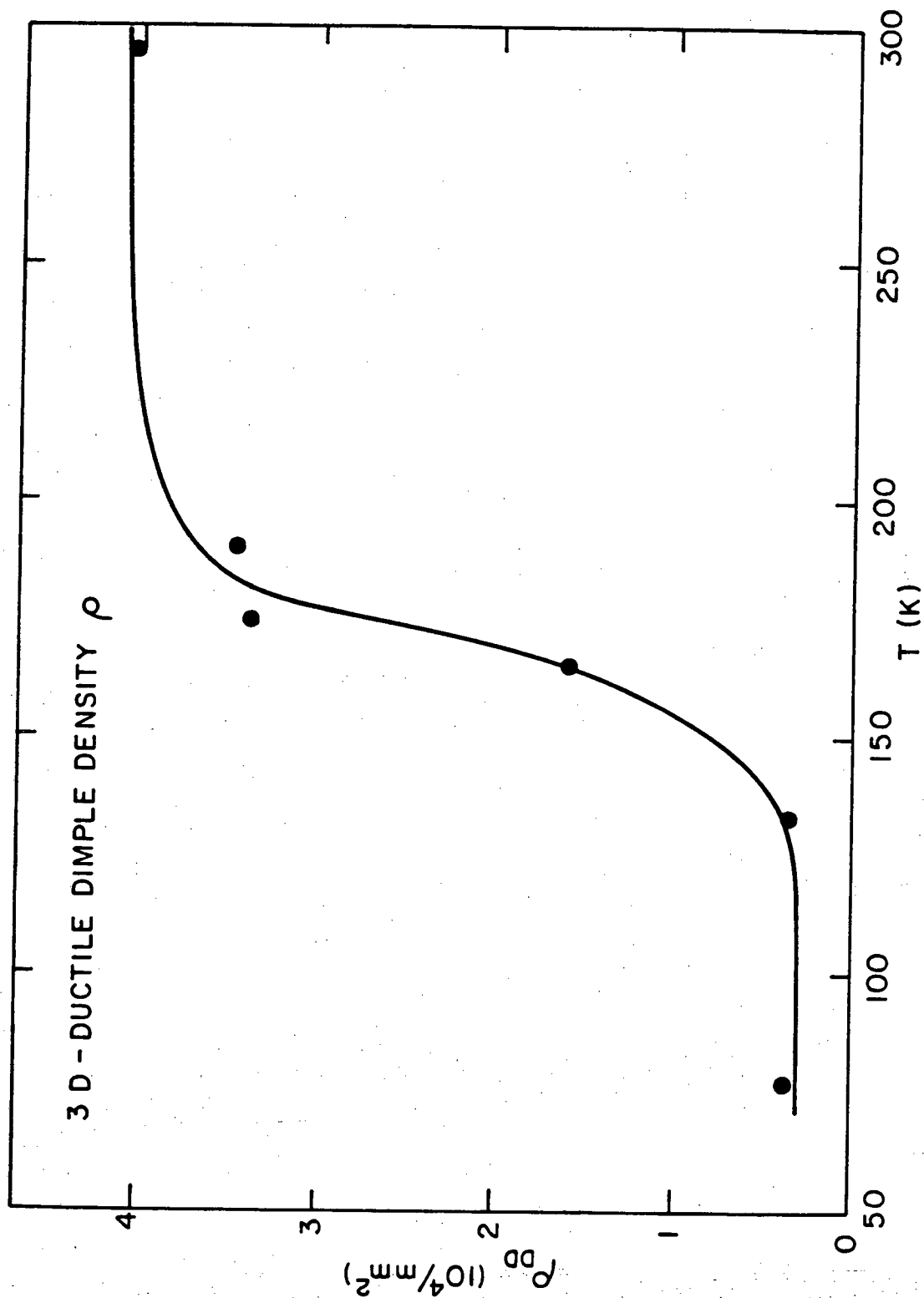
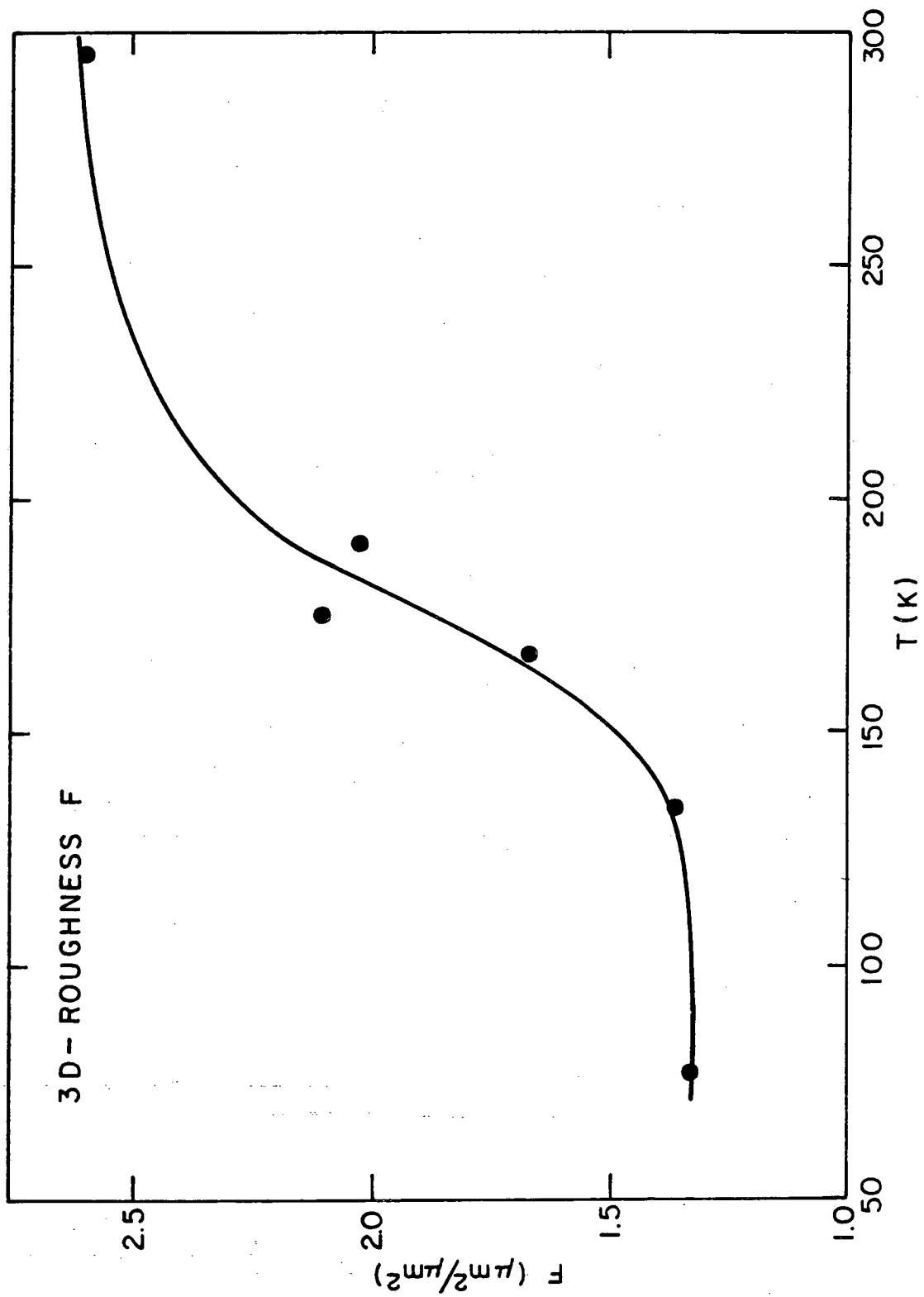
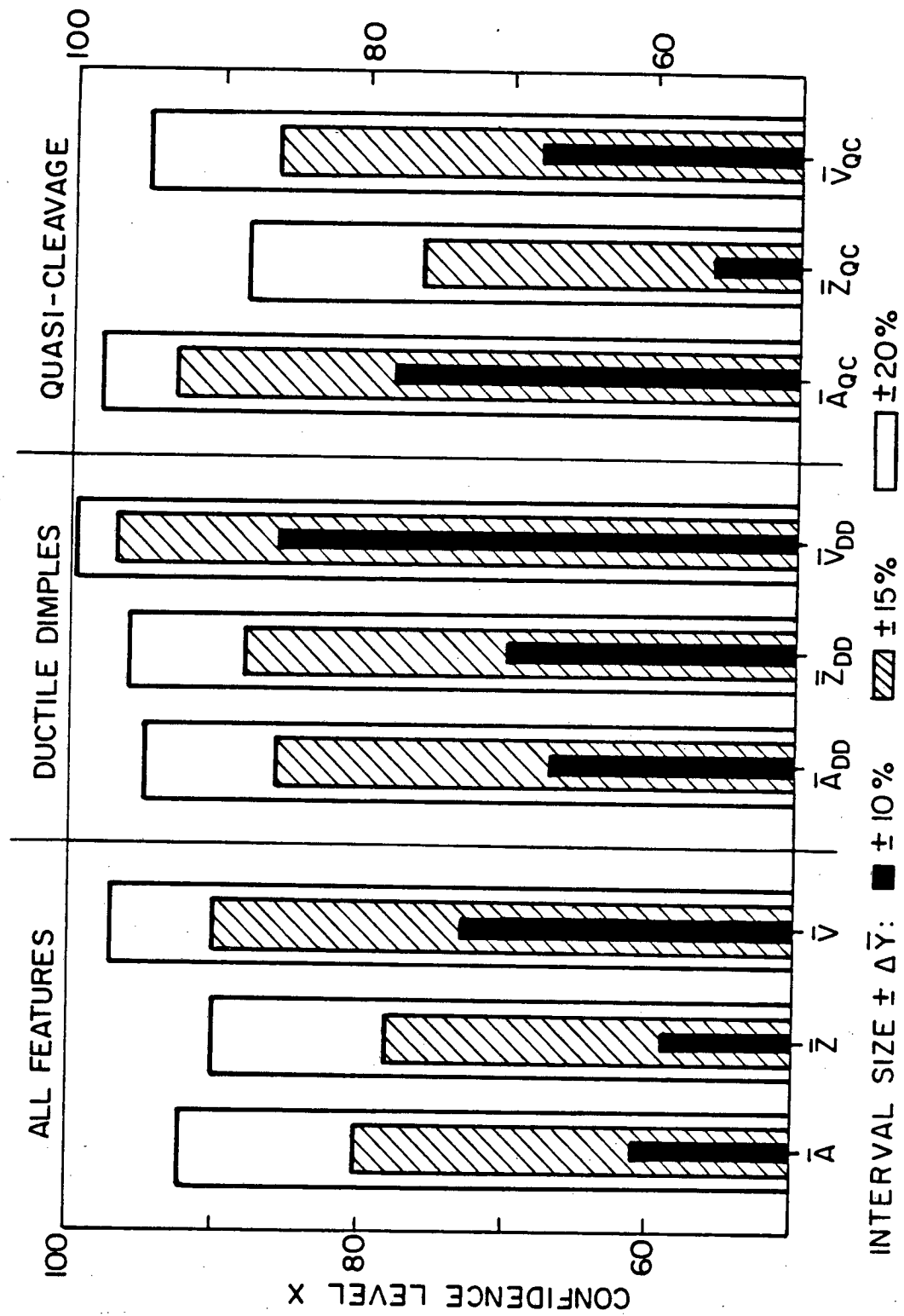


Figure 24



XBL 8110-6899

Figure 25



XBL 823-5455

Figure 26

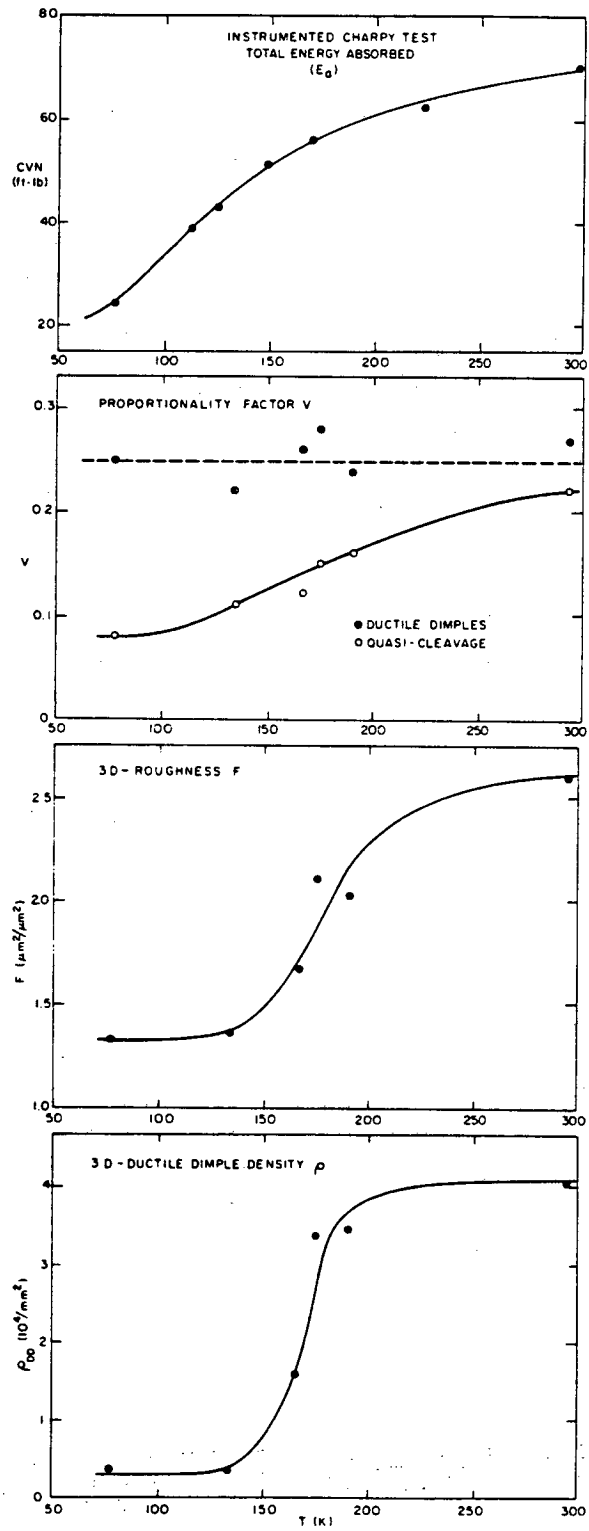


Figure 27

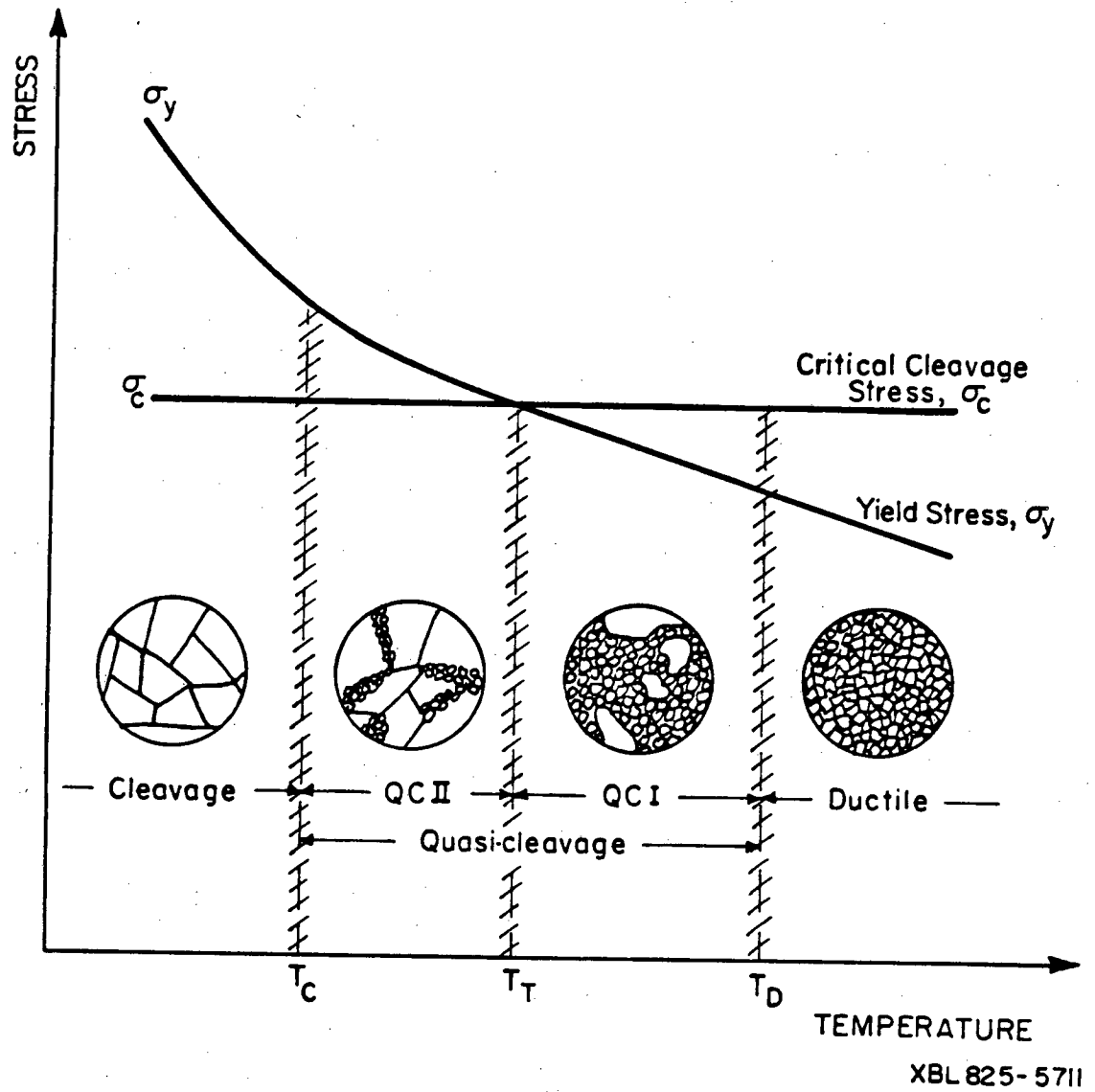


Figure- 28

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