VOLUME FRACTION DETERMINATION IN CAST SUPERALLOYS AND DS
EUTECTIC ALLOYS BY A NEW PRACTICE FOR MANUAL POINT COUNTING

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16. Abstract

Volume fraction of a constituent or phase was estimated in six specimens of conventional and DS-eutectic superalloys, using ASTM E562-76, a new "Standard Recommended Practice for Determining Volume Fraction by Systematic Manual Point Count". The Practice will appear in the Annual Book of ASTM Standards. Volume fractions determined ranged from 0.086 to 0.36, and with one exception, the 95 percent relative confidence limits were approximately 10 percent of the determined volume fractions. Since the confidence-limit goal of 10 percent, which had been arbitrarily chosen previously, was achieved in all but one case, this application of the new Practice was considered successful.

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SUMMARY

A new, broadly applicable, "Standard Recommended Practice for Determining Volume Fraction by Systematic Manual Point Count", ASTM E562-76, was evaluated in some practical applications by using it to estimate volume fraction of a constituent or phase in six nickel and cobalt superalloy specimens. These were developmental alloys for aircraft turbine blades. They included two specimens each of two similar conventionally-cast superalloys, and one each of two directionally solidified (DS) eutectic alloys, one rod type and one lamellar type.

Volume fractions for "gamma-prime eutectic nodules" of the four conventionally-cast superalloy specimens ranged from 0.11 to 0.14; 95-percent relative confidence limit (RCL) of the volume fraction was approximately 10 percent for three of the specimens and 17 percent for the fourth. Volume fraction of reinforcing phase for the rod eutectic was 0.086; for the lamellar eutectic it was 0.36. Corresponding RCL values were 9 and 8 percent. Volume fractions for the latter two alloys determined by a television-type scanning instrument were higher. This probably resulted from a combination of visual bias and instrument bias in establishing the signal level corresponding to the edge of a feature. RCL values for the instrument-determined volume fractions were approximately 40 percent lower (better) than those for the manually-determined volume fractions, as expected from the very large number of "points counted" by the instrument.

The Recommended Practice was found to be operationally feasible. The volume fractions obtained for the DS eutectic alloys were compared with literature values for similar microstructures, and found to be reasonable. Relative confidence limits of the volume fractions indicated that in these micro-

structures the matrixes and second phases formed nearly "ideal Poisson mixtures".

INTRODUCTION

Many materials specifications include an upper (and/or lower) limit for the volume fraction of one or more specific phases or constituents. Determination of volume fraction for this purpose by a semi-automatic or automatic instrument is usually acceptable, even desirable. However, such equipment frequently is not available, so that manual methods must be used. Also, certain types of microstructural features do not show sufficient or consistent-enough contrast to permit their being analyzed or characterized by any known instrument. Moreover, if an instrument is operating incorrectly or is not calibrated frequently, or if feature-edges are not set correctly, the user may unknowingly obtain consistently low or high (i.e., biased) volume fraction values, relative to those from correctly conducted manual point counting.

Systematic point counting has been shown to provide a more precise estimate of volume fraction for a given amount of work than any other manual method.[1] However, a standardized procedure for conducting point counts has not been available. Such a procedure has now been prepared by ASTM Committee E4 (Metallography) and will appear soon in The Annual Book of ASTM Standards as E562-76, a Standard Recommended Practice (RP). It is intended to be applicable to a broad range of macro and microstructures. It includes provisions for estimating, after a few preliminary measurements, the amount of point counting required for a desired confidence limit of the result. It also provides for subsequently calculating from the point counting results, both the volume fraction (V_V) and its 95 percent relative confidence limit (RCL; i.e., confidence limit of V_V expressed in percent of V_V). The general features of the Recommended Practice have been described in a paper by R. Millsop.[2]

The geometric principles on which systematic manual point counting is based have been applied for over 100 years in many disciplines without evidence of any consistent bias in the results obtained.

The experiments described here were undertaken to learn how well the stepwise procedures of the new RP would work when applied to point counting the microstructures of some available developmental alloys. Volume fraction and its RCL were determined for each of six specimens of experimental cast nickel and cobalt base superalloys. One deviation from the detailed stepwise instructions of the RP was made to minimize the labor of laying out additional point counting locations on the specimens. The deviation is explained under Materials, Apparatus and Procedure. While it would not be allowable in many materials-acceptance situations, the deviation was found to have minimal effect on the results of this study.

The RP was applied to estimation of V_V in six metallographic specimens of experimental nickel and cobalt base alloys, which were prepared in programs aimed at development of improved materials for aircraft gas turbine blades. The volume fraction information was desired as part of characterization of the microstructures for the experimental alloy programs. Results from the specimens were examined for precision and reasonableness and compared for consistency with one another. However, since satisfactory alternate manual methods for estimating V_V are less fundamental than systematic point counting and are costly and time-consuming, the question of accuracy was not examined directly.

The study described included use of a 9-point eyepiece grid, applied systematically to several microstructures using a conventional light metallograph. It also included use of a 100-point, 7-cm-square grid manually positioned on each of 50 scanning electron micrographs, for a microstructure which could not be resolved adequately by light microscopy.

Only two of the six specimens had microstructures of suitable contrast for volume fraction determination by a semi-automatic scanning instrument (quantitative television microscope). For these specimens, volume fractions and their RCL's were obtained by this method (which can be considered as point counting with an extremely dense grid), and have been compared with those from manual point counting.

MATERIALS, APPARATUS AND PROCEDURE

Materials

Processing and compositional information is given in Table I for the two conventionally cast superalloys (two specimens of each) and the two directionally solidified (DC) eutectic alloys used to evaluate the proposed Recommended Practice (RP). The two conventionally cast, nickel-base superallows were similar except for small but metallurgically significant differences in composition. Both exhibited typical gamma/gamma-prime microstructures (Fig. 1). The cobalt-base DS eutectic microstructure consisted of a gamma solid solution reinforced by hafnium carbide (HfC) rods (Fig. 2(a), and the nickel-base eutectic microstructure contained alternate lamellae of gamma solid solution and (Fe, Ni)Al intermetallic phase (Fig. 2(b). All specimens were encapsulated in bakelite metallographic mounts and were polished and etched by conventional procedures.

Apparatus

The manual point counting apparatus included a light metallograph, a scanning electron microscope, a 100-point test grid and a 9-point test grid. The 100-point grid was 70 mm square on clear plastic, with the exact crossing points of the lines left clear, as suggested in the Recommended Practice. It was used for point counting 90 by 100 mm scanning-electron photomicrographs. The 9-point square grid was part of a commercial grain-size eyepiece, and was used for point counting directly on the specimen in the light metallograph. Both grids are illustrated in Fig. 3.

Non-manual, semi-automatic determinations of volume fraction (V_V) were made using a television-type scanning instrument, the Quantimet B. It was equipped with a macro stage and a metallographic microscope, both of which were used in this study.

Procedure for Manual Point Counting

The procedural steps for systematic manual point counting are described
in the Recommended Practice (RP) and are not detailed here. However, they
can be visualized as three sequences of applying the test grid to the micro-
structure and conducting the point counting, plus the required calculation:
The first sequence provides a rough estimate of volume fraction from a very
few point counts, for use in estimating how many total grid points need to be
used in the main point counting process, to obtain V_V with the desired con-
fidence limit. The second and third sequences form the two halves of the main
point counting process. The spread of the volume fraction values (i.e., the
magnitude of the relative confidence limit, RCL) from the second sequence
(first "half" of main point counting) is used to decide whether to increase, de-
crease, or leave unchanged, the amount of point counting to be done in the
third sequence (second "half"). Volume fraction values are calculated from
the results of the first, second, and second-plus-third sequences, with each
value being refined relative to the preceding one. The 95 percent relative
confidence limit is calculated for the volume fraction values of the second, and
second-plus-third sequences only.

In the work described, a desired relative confidence limit was first se-
lected, as called for by the Recommended Practice; 10 percent was arbitrarily
chosen. An ideal Poisson distribution of second-phase (alpha) features was
assumed, as described in section 9.1.4.2 of the RP (Appendix A of this re-
port). Corresponding to this 10 percent RCL (column 2 of Table AI, Appen-
dix A), row 2 of Table AI was used for extimating the amount of point counting
to be done in the second and third sequences, after the first sequence had been
completed. However, before the first sequence for each specimen was started,
possible arrays of test locations also were considered, based on both the
available, suitable specimen area, and the feasible specimen-stage translation
increments which would be compatible with the graduations on the stage controls.

The first sequence was then carried out by applying the test grid to the
microstructure a few times, counting the alpha hits, and calculating a pre-
liminary value of volume fraction. Row 2 of Table I of the Recommended
Practice (Table AI of Appendix A) was used to determine how many total grid

points should be required, and from that number, what array of locations on the specimen should be feasible, to provide the desired confidence limit. See section 10.1 of Appendix A.

In the next steps, a deviation was made from the procedures of the Recommended Practice (section 10.2, Appendix A). All of the locations of the selected array were point counted without an interruption at the midpoint to evaluate progress, in effect combining the second and third sequences. To have made the evaluation and adjustment at the midpoint as called for, with uniform sampling of the available specimen area, one would have needed to carefully redesign the array of locations to be point counted subsequently (third sequence). This would have required more time than was considered justified.

To avoid area-selection bias during the point counting, the specimen stage was translated to each location of the array in succession by incrementing the stage controls the same fixed, precalculated number of graduations for each position change. The specimen image was not observed during the translations. This assured that the point counting was in fact "systematic". During counting at each location, grid points falling on or doubtfully close to interfaces between the phase of interest ("alpha") and the matrix were counted as 1/2 point.

To partially compensate for the deviation in procedure indicated above, the midpoint calculations of V_V and RCL were carried out "after the fact" (after all of the point counting had been done) in conjunction with the corresponding final calculations. For all but two of the specimens examined, this exercize showed that no adjustment at the midpoint of the amount of point counting required subsequently would have been necessary. Of the two exceptions, according to these "after the fact" intermediate RCL values, one specimen would have required no point counting after the intermediate check, and the other would have required approximately three times as many points to be counted after the "midpoint" (twice as many total grid points) as originally estimated, to yield the desired 10 percent relative confidence limit.

The four conventionally cast superalloy specimens and the lamellar DS eutectic specimen were manually point counted directly in the microscope using the 9-point eyepiece grid. For all four of the conventionally cast spec-

imens, a 17- by 17-location array was used, yielding a total of about 2,600 grid points applied and counted. For the lamellar DS eutectic specimen, a relatively small fully-lamellar region was available, so that a smaller 7- by 21-location array was used, giving about 1,300 total grid points.

Because the microstructure of the rod eutectic specimen was very fine, scanning electron micrographs were needed to resolve it. Fifty 1,300 X micrographs were taken at a 10- by 5-array of locations on the specimen. Each micrograph was systematically manually point counted by centering the 100-point macro-grid on it and counting the alpha "hits".

Procedure for Volume Fraction by Scanning Instrument

Volume fractions and their relative confidence limits for the two DS eutectic alloy specimens were determined by the scanning-type semi-automatic instrument, as well as by the manual point counting RP. (The four conventionally cast superalloy specimens could not be etched with sufficient contrast between alpha features and matrix to permit use of the instrument to determine volume fraction.) Seventy systematically laid-out fields of the lamellar DS eutectic specimen were examined directly by the instrument, using the image from the "built-in" metallographic light microscope. The finer structure of the rod eutectic specimen required that the fifty scanning electron micrographs previously prepared for use in manual point counting be examined by the instrument, rather than the specimen itself. The macro stage and illuminator system of the instrument was used for this purpose.

The RP was developed for manual point counting, and is not intended to apply to determination of V_V and RCL by instrument. However, for convenience, and in order to compare instrument results with manual point counting results, the data-organization and calculation steps of the RP were used in calculating V_V and RCL values. Here as in the manual point counting, all locations of the selected array (70 locations for the lamellar eutectic and 50 for the rod eutectic) were "counted" by the instrument without either a preliminary V_V estimate or a midpoint check of progress. However, again "after the fact" as in the case of the manual point counting, V_V was recorded for the first field (first sequence, or "preliminary estimate"), for the first half of the fields

(second sequence), and for all of the fields (second plus third sequences) examined by the instrument. Similarly, the RCL of V_V for the first half of the fields, and for all of the fields, was calculated and recorded. The values of V_V for each of the 70 locations of the lamellar eutectic were organized into 10 subgroups of 7 values each, and the corresponding 50 V_V values for the rod eutectic were organized into 10 subgroups of 5 values each. The meter of the scanning instrument was calibrated to read V_V directly for each field of view examined by it.

The measured area of the instrument's television image may reasonably be assumed to contain 60,000 picture points (250 scan lines, squared). Therefore, 60,000 can be taken as the number of "grid points applied" to each field of view of a microstructure and "point counted" by the instrument. This number, multiplied by the number of fields examined in each DS eutectic specimen, gives an equivalent "total number of grid points applied to the microstructure and counted" by the instrument. The latter number was used in comparing instrument results with results from manual point counting of the same specimens. For the lamellar eutectic it was 4.2 million "grid points"; for the rod eutectic, 3 million.

RESULTS AND DISCUSSION

Volume fraction and relative confidence limit results are shown in Table II and Fig. 4. The volume fraction (V_V) values are given for each specimen, corresponding to the preliminary-estimate, intermediate (midpoint) and final point count results. Relative confidence limits (RCL) are shown for intermediate and final results. Total numbers of grid points, both estimated and actually applied and counted, are also shown for each specimen. Corresponding results for volume fraction determinations made with the semiautomatic scanning instrument are also given in Table II.

Manual Point Counting Results

It is evident from the V_V data of Table II, plotted in Fig. 4, that the final volume fractions of the four conventionally cast superalloy specimens were

closely grouped in the narrow range 0.11 to 0.14. Figure 4 also shows that as the amount of point counting increased, from preliminary through intermediate to final results, the corresponding volume fraction values for these four specimens converged toward a common value of about 0.12. This suggests that the four specimens really represented a single statistical population with respect to volume fraction of gamma-prime eutectic nodules.

The final volume fractions for the two directionally solidified (DS) eutectics were widely different, as expected with two different types of eutectic microstructures - rod and lamellar. V_V for the tantalum-carbide rods in the cobalt-base alloy was 0.086, and for the (Fe, Ni)Al intermetallic-phase lamellae in the nickel-base alloy it was 0.36. Confidence in the accuracy of the manual point counting method resulted from the fact that the foregoing volume fractions determined for the two DS eutectic specimens were within the ranges reported in the literature for similar DS eutectic microstructures. For rod eutectics, literature values ranged from 0.057 to 0.087 (3), compared to the value of 0.086 reported here. For lamellar eutectics, literature values were in the range 0.32 to 0.43 (4); the value reported here is 0.36.

As previously indicated, a desired relative confidence limit of 10 percent was arbitrarily selected for the point counting reported here. It was also arbitrarily assumed that the materials examined had distributions of alpha features and matrix approaching those of Poisson mixtures ("ideal" RCL). These two considerations led to use of Table AI (Appendix A), row 2 (10 percent ideal relative confidence limit) for the decision of how many (total) grid points should be applied to each specimen microstructure and counted. In general, it was found that when approximately the number of grid points called for by Table AI had been applied and counted, the goal of an RCL of 10 percent (or less) had indeed been achieved. From these results it was concluded that the materials examined did approximate Poisson distributions of alpha phase and matrix.

It has been common experience that volume fractions of second phase in most commercial metals, determined by point counting, exhibit relative confidence limits 1.5 to 3 times as broad as those of corresponding Poisson distributions. This is the basis for including column 3 of Table I of the RP (Table AI, Appendix A of this report), which lists for each row a range of RCL values 1.5 to 3 times as broad as the corresponding value for column 2

("ideal" RCL).

Three of the four conventionally cast superalloy specimens exhibited a final relative confidence limit of 10 percent. The RCL of the fourth one was distinctly broader at 17 percent, indicating that it had a less uniform microstructure than the other three. The relative confidence limits for the rod and lamellar DS eutectics were 9 and 8 percent, respectively, indicating that these microstructures were even more uniform than those of the three conventionally cast superalloys which had 10 percent RCL's. This is not surprising, since the unique and closely controlled solidification conditions required to grow DS eutectics would be expected to provide unusually uniform microstructures within the aligned portions of the cast bars.

The rod eutectic with its relatively low "preliminary V_V estimate" of 0.08, was "predicted" by Table AI to require 5,800 total grid points applied for an RCL of 10 percent. The 5,000 points actually used resulted in an RCL of 9 percent. On the other hand, the lamellar eutectic, with the much higher preliminary V_V estimate of 0.42, should have required only 1,000 total grid points for an RCL of 10 percent. The 1,300 points actually used resulted in an RCL of only 8 percent. The results were reasonably close to the predictions for both of these specimens.

All of the relative confidence limits decreased (i.e. the distribution became narrower), or remained unchanged, from intermediate to final values (Table II and Fig. 4). A decrease is to be expected, since ideally for systematic manual point counting, the ratio of final RCL to intermediate value should be 0.7 (square root of 0.5, the ratio of the two amounts of counting done). However, considerable scatter is usually encountered. For the present results, this ratio varied between 0.5 and 1.0.

Viewed slightly differently, statistical analysis of the manual point counting results indicated that for four of the six specimens, the number of grid points actually applied and counted was within 30 percent of the number predicted by the intermediate RCL results to be needed. Of the two exceptions, for one specimen (conventionally cast superalloy specimen 645A), over twice as many grid points were used as were subsequently predicted necessary from the intermediate RCL results. Yet the final RCL was 10 percent, rather than a smaller value as might have been expected. It is probable that the region of

the specimen examined during the first half of the point counting was more uniform in microstructure than that examined during the second half.

The second exception was conventionally cast specimen 646A, for which the final RCL was 17 percent. The total number of grid points used was approximately 40 percent less than the number predicted from the intermediate RCL to be needed. The excessive final RCL of 17 percent in this case verified the prediction resulting from the high intermediate RCL (23 percent), that the total number of grid points actually used, 2,600, was too low to yield a V_V value with the desired RCL of 10 percent. Apparently the microstructure of this specimen was consistently less uniform than the microstructures of the other three conventionally-cast superalloy specimens.

Volume Fraction Results by Scanning Instrument

For both of the DS eutectic alloys, the values of V_V obtained with the semiautomatic scanning instrument were essentially unchanged from the intermediate results to the final results. V_V was 0.09 for the rod eutectic, and 0.45 for the lamellar eutectic. For these two specimens, the constancy of V_V with increasing number of measurements is probably related to both the uniformity of the DS eutectic microstructures, and the very large number of "grid points" examined by the scanning instrument in each field of view.

According to the Hilliard condition[5] (section 8.1 of the RP: Appendix A here), most of the very large number of "total grid points applied and counted" by the scanning instrument were redundant (see last paragraph of "Materials, Apparatus and Procedure" section). Moreover, since the density and total number of grid points were much larger than with manual point counting, so that no alpha features in any field examined were missed by the instrument "counting", the variability of the results approached the variability of the material as a limit. That is, the proportion of the RCL which resulted from variability of the measuring process became negligibly small. Therefore, the RCL values for the instrument-determined V_V values were not reduced, relative to the manual-point-count RCL's, in proportion to the greatly increased number of "gridpoints" used by the instrument. However, the instrument RCL's were still only 1/2 as large as the manual RCL's.

The V_V values obtained by the scanning instrument on the DS eutectic alloys were higher than the corresponding V_V values from the manual point counts. The instrument V_V for the rod eutectic was 8 percent higher than the manual value; for the lamellar eutectic the instrument value was 25 percent higher than the manual value. The differences are believed to be due to a combination of (a) a tendency for the semi-automatic instrument circuitry to bias results toward high V_V values (i. e., to "overcount"), and (b) insufficient sharpness of the television image to permit the operator to set the detection levels for edges of alpha features with reasonable accuracy, at least at the image magnifications used here.

The ratio of final-volume-fraction RCL to intermediate-volume-fraction RCL for the two scanning instrument determinations was 0.5 for the rod eutectic and 1.0 for the lamellar eutectic. The corresponding ratios for the manual-point-count RCL's were 0.5 and 0.6, respectively.

Evaluation of the Recommended Practice

The work of systematic manual point counting consists mainly of (a) laying out the array of grid-placement locations and point counting each location, and (b) calculating volume fractions (V_V) and relative confidence limits (RCL). The time required for (a) was found to be in the order of two hours. Organizing and calculating results, using a hand calculator, required another 1 to 1.5 hours. These times of course would be reduced or increased, respectively, by broader or narrower confidence limit requirements, or higher or lower volume fractions encountered. However, it seems likely that for similar V_V levels and precision (RCL) to that sought here, with increasing operator experience and skill, the time for point counting according to the Recommended Practice could be reduced to perhaps 1 hour. Similarly, the data handling and processing time could probably be reduced to 0.5 hour or less. In general, as indicated by Table AI, the amount of point counting required (number of grid points applied) is quadrupled when the required RCL is decreased by 1/2. Furthermore, a decrease of 1/2 in the volume fraction of alpha present will double the amount of point counting required for the same RCL. (It should be noted in Table AI, Appendix A (column 4) that for a given

desired precision, the estimated number of grid points <u>falling on alpha phase</u> is constant regardless of the volume-fraction level encountered in a specimen.)

In general, the results described here, obtained in practical use of the ASTM Recommended Practice (RP), have been encouraging. However, the new user of the RP is likely to find that the initial learning stages are tedious. He will need to read the Recommended Practice carefully and operate with meticulous attention to detail. On the other hand, as familiarity and speed are gained in applying the various steps and precautions, the tedium should be largely displaced by interest and satisfaction in the results obtained. It is anticipated that a normally skilled technician will quickly become proficient in using it. With the prescribed precautions to avoid bias, he should obtain V_V values which are accurate and precise.

SUMMARY OF RESULTS

A new ASTM "Standard Recommended Practice for Determining Volume Fraction by Systematic Manual Point Count" was application-tested on six developmental nickel and cobalt-base-alloy specimens. These included four specimens of two conventionally-cast superalloys and two specimens (one each) of two directionally solidified (DS) eutectic superalloys. For comparison purposes, volume fraction of the two DS eutectic alloys was also determined by means of a semi-automatic scanning instrument. Major results for the manual point counting were as follows:

1. The Recommended Practice was found to work well, and the volume fractions and their relative confidence limits obtained using it were reasonable relative to values for "ideal Poisson" materials given in the Recommended Practice.

2. Final volume fraction (V_V) values for the gamma-prime eutectic con- stituent in the conventionally cast superalloy specimens ranged from 0.11 to 0.14. Final relative confidence limit (RCL) for three of the specimens was 10 percent; for the fourth it was 17 percent. The latter high value has been attributed primarily to an exceptionally inhomogeneous microstructure in that particular specimen.

3. By manual point counting, the final volume fraction of reinforcing phase for the rod DS eutectic was 0.086; for the lamellar DS eutectic it was 0.36. These values are consistent with ranges of volume fraction values reported in the literature for similar DS eutectic structures. The corresponding RCL's for these two alloys were 9 and 8 percent, respectively.

4. In every case, the RCL for the intermediate V_V result (when approximately 1/2 of the anticipated point counting had been done) was equal to or larger than the RCL for the final V_V result, as would normally be expected. (Ideally the final RCL is 0.7 times the intermediate RCL: here that ratio ranged from 0.5 to 1.0.)

5. The volume fractions for the two DS eutectics obtained by the semiautomatic scanning instrument were higher than those from the manual point counting, possibly as a result of instrument characteristics, and operator uncertainties in setting detection levels for edges of alpha features. The RCL's for the instrument V_V values were smaller than the corresponding manual point count RCL's, by a factor of approximately 0.4. Much larger decreases would be expected as a result of the very large number of effective "grid points" (mostly redundant) applied to the microstructures and "point counted" by the instrument scanning and detection systems. However, the final RCL values obtained probably could not be decreased further significantly, because they represented the inherent RCL's (variabilities) of the microstructures themselves, without significant contribution from variability of the measuring process.

APPENDIX A

Excerpts From ASTM E562-76, "STANDARD RECOMMENDED PRACTICE FOR DETERMINING VOLUME FRACTION BY SYSTEMATIC MANUAL POINT COUNT", Referred To In This Paper

9.1.4.2 - It has been assumed that the necessary amount of point counting to estimate volume fraction in a structure within a given confidence limit, can be predicted by Poisson statistics (provided that the Hilliard and antimoire' conditions are observed; see 7.3, Section 8, 9.1.3.2, and 9.1.3.3).

All of Table AI except column 3 was calculated based on this assumption. This assumption is in turn totally dependent on the assumption that the material is in fact a Poisson mixture. Such a mixture is by definition uniform in composition when sampled by any array of large samples, but the exact location of each alpha feature is locally random. As a result, small samples exhibit the specific statistical characteristics of the Poisson distribution.

10.1 - <u>Preliminary Estimate of Grid Points to be Examined</u> -- To obtain the preliminary estimate, apply a suitable grid to the structure several times in different areas and count the alpha hits. Then divide the sum of these counts by the total number of grid points to obtain an approximate volume fraction of alpha, and then convert to percent. Referring to Table AI, use this value to select which of Columns 5 through 10 apply, interpolating where necessary. Then from Column 3 select the row corresponding to the desired range of precision (RCL). From the volume percent and precision values, Table AI gives the total number of grid points which Poisson statistics predict should be applied to the structure and examined to obtain the desired precision. Note that Table AI gives grid points, which is equal to the number of gird applications multiplied by the number of points in the test grid.

10.2 - <u>Intermediate Check of Progress</u> (Second Estimate of Grid Points to be Examined) -- When about half of the predicted point counting is done, analyzed the data collected. This will provide a more realistic prediction than that of 10.1 of the total number of grid points that should be examined.

10.2.1 - First, add up the total points on alpha phase at this stage. If the total is less than half of the required ΣP_α (column 4 of Table AI), it is already apparent that more grid applications are required than first predicted (10.1).

10.2.2 - Next, organize the data and make a statistical analysis as outlined in Section 11. If the RCL is greater than 1.4 times the desired RCL, it will be necessary to increase the number of grid points applied (from the estimate made in accordance with 10.1) by the ratio of the square of the RCL just determined to two times the square of the desired RCL.

10.2.3 - Using the new number of total grid point applications required (Section 10), revise the array of locations at which the grid is to be placed and examined to provide as uniform coverage as possible of the available specimen

area. Then carry out the steps and precautions outlined in sections 7.2 through 7.5 for grid applicatio \ counting, and recording until the required number of grid points has been applied to the structure. See also Annex A1.

8.1 - The Hilliard condition[5] recognizes that variance of the observations results both from variation of the number of alpha features or grains between fields and variation of the manner in which the test points fall on the fields tested. Both being basically Poisson, the precision depends on the number of alpha features and on the number of points on alpha in the field tested, with the smaller of these numbers dominant. Under the Hilliard condition, many alpha features are not hit by points, so that the number of points on alpha mainly controls. With a very dense raster, as in machine scanning, the number of features controls, but the standard error usually can be reduced by no more than 30 percent. The Hilliard condition thus provides acceptable precision with minimum labor.

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TABLE AI. - PREDICTED MINIMUM NUMBER OF GRID POINTS TO ESTIMATE VOLUME

FRACTION, V_V, AS A FUNCTION OF DESIRED PRECISION

Step 1. - From column 2 or 3, select a row i (1 - 4) that gives the desired relative precision, RCL.

Step 2. - From columns 5 - 10, select a column j that reflects the preliminary estimate of $V_V(\alpha)$.

Step 3. - Intersection of row i and column j gives the minimum number of total grid points, which Poisson statistics predict it is necessary to apply to the microstructure and count to attain the selected precision (Step 1).

Column	1	2	3	4	5	6	7	8	9	10
Row	Desired percent presicion[a] (Relative)[b]			Points on phase, ΣP_α	Total points in all grid applications, ΣP_T at percents of second phase indicated					
	Ideal		Expected (real struc) CL range							
	CV	CL			1% α	2% α	5% α	10% α	20% α	40% α
1	2.5	5	7.5 - 15	1,600	160,000	80,000	32,000	16,000	8,000	4,000
2	5.0	10	15.0 - 30	400	40,000	20,000	8,000	4,000	2,000	1,000
3	10.0	20	30.0 - 60	100	10,000	5,000	2,000	1,000	500	250
4	20.0	40	60.0 - 120	25	2,500	1,250	500	250	125	60

[a]Explanation of Precision - an ideal aggregate of uniform composition with particles randomly positioned, when ideally sampled, is expected to conform to Poisson statistics, for which

$\sigma_{P\alpha} = \sqrt{P_{\alpha'}}$ with P_α being the number of counted particles (features) of the second (minor) (α) phase. The Coefficient of Variation, $CV = \sigma_{P\alpha}$, depends only on the number of counted points on α phase, again for the assumed Poisson distribution, which is used only for predicting the number of grid points which are required to be counted, for a given precision. From this CV, the ideal relative confidence limit (Precision), RCL (at 95% confidence level) can be determined. Real observations will not attain ideality, and may be expected to demonstrate (by observed variation) larger confidence limits. These larger confidence limits are the values indicated in column 3 (essentially, 1 1/2 to 3 times RCL-ideal).

[b]The precisions indicated in column 1 - 3 are relative to the actual volume fraction or percent α determined. For example, if a relative confidence limit (RCL) of 10% is demonstrated for a specimen yielding 10% (volume) α, the result may also be reported as $V_V = 10\pm1\%$.

TABLE I. — ALLOYS USED FOR EVALUATING THE PROPOSED ASTM

RECOMMENDED PRACTICE FOR DETERMINING VOLUME FRACTION

BY SYSTEMATIC MANUAL POINT COUNT

Alloy/Specimen Designation	TRW-NASA VIA, Modified		Kim no. 80	SNT-9D
	L645A & L645B	L646A & L646B		
Casting Method/Type	Conventional	Conventional	DS/Rod Eutectic	DS/Lamellar Eutectic
Processing	As investment cast plus annealed 926° C 16 hr		As direction-ally solidified at 0.8 cm/hr; Gradient = 250° C/cm	As direction-ally solidified at 1 cm/hr; Gradient = 100° C/cm
Test section	See footnote	See footnote	Transverse, entire 12 mm diameter bar	Transverse, entire 12 mm dia. (out-of-round) bar
Approximate composition, wt %				
Ni	Bal	Bal	20	Bal
Fe	----	----	--	34
Co	7	7	Bal	----
Cr	8	8	15	9
HfC	----	----	11	----
Ta	7	6	--	----
Al	6	5	--	8.5
W	4	6	--	----
Mo	3	2	--	----
Ti	1	1	--	----
Hf	0.8	0.7	--	----
Cb	.6	.5	--	----
Zr	.3	.1	--	----
C	.1	.1	--	----
B	.03	.03	--	----

Footnote: Specimens L645A and L646A were machined from cast and annealed rec-
tangular blocks 3.8 cm × 7.6 cm × 17.8 cm (1.5 in. × 3 in. × 7 in.). Specimens
L645B and L646B were machined from preform castings only slightly larger than
the finish dimension of 1/4 in diameter (gage section). In each case, therefore,
the A specimen had a coarser microstructure than the B specimen.

TABLE II. - RESULTS OBTAINED IN USING THE PROPOSED RECOMMENDED PRACTICE TO ESTIMATE VOLUME FRACTION IN SIX

NICKEL AND COBALT-BASE ALLOY SPECIMENS

(A required precision, i.e., 95% relative confidence limit of Volume Fraction, V_V, of 10% was used, with alloys examined assumed to be ideal "Poisson mixtures")

Material	Specimen number	Preliminary estimate		Intermediate result and new estimate				Final results		
		V_V	Approximate total grid points req.[a]	Total points applied thus far	Volume fraction, V_V	Rel. conf. limit, RCL, %	New total points req. per sec., 8.2	Total grid points applied	V_V	Rel. conf. limit, RCL, %
Superalloy	L645A	0.18	2,400	1,224	0.15	10	1,200	2,600	0.14	10
"	L645B	.18	2,400	1,224	.14	15	2,700	2,600	.13	10
"	L646A	.092	4,600	1,224	.089	23	6,350	2,600	.11	17
b "	L646B	.15	3,000	1,224	.12	16	3,070	2,600	.12	10
Rod eutectic	KIM80	.077	5,800	2,500	.082	18	6,560	5,000	.086	9
Lamellar eutectic	SNT9D	.42	1,000	600	.38	13	1,040	1,323	.36	8
Rod eutectic	KIM80	.081	Not applicable	1.5×10^6[d] 25e	.090	11	Not applicable	3.5×10^6[d] 50e	.093	5[f]
c Lamellar eutectic	SNT9D	.43		2.1×10^6[d] 42g	.45	5		4.2×10^6[d] 70g	.45	5[f]

[a] See Table I of proposed Recommended Practice, Appendix A

[b] By manual point counting

[c] By semi-automatic scanning instrument

[d] "Total grid points applied", based on an assumed 60,000 "picture points" of scanning instrument

[e] Number of micrographs measured by instrument

[f] Relative confidence limit (RCL) based on the V_V value for each field (or micrograph) being treated as a subgroup. V_V is read directly on instrument meter for each field of view

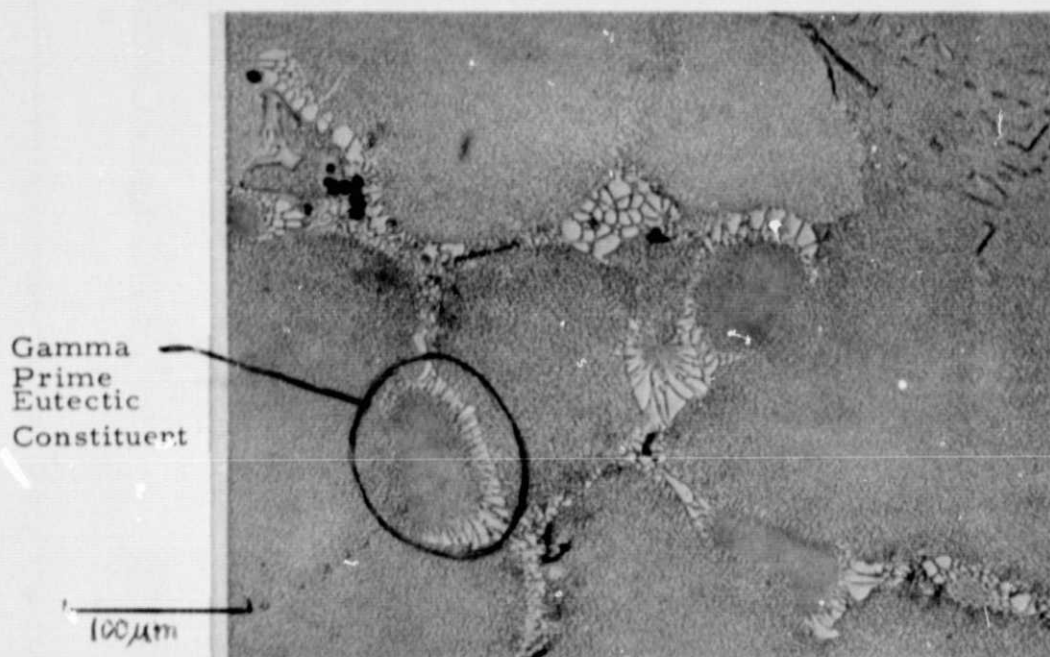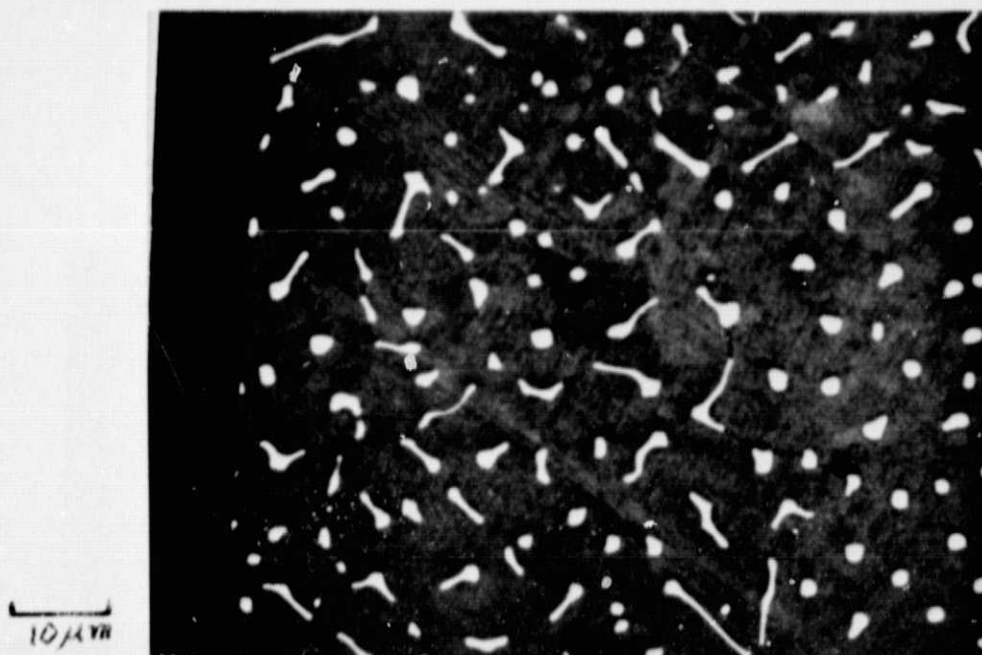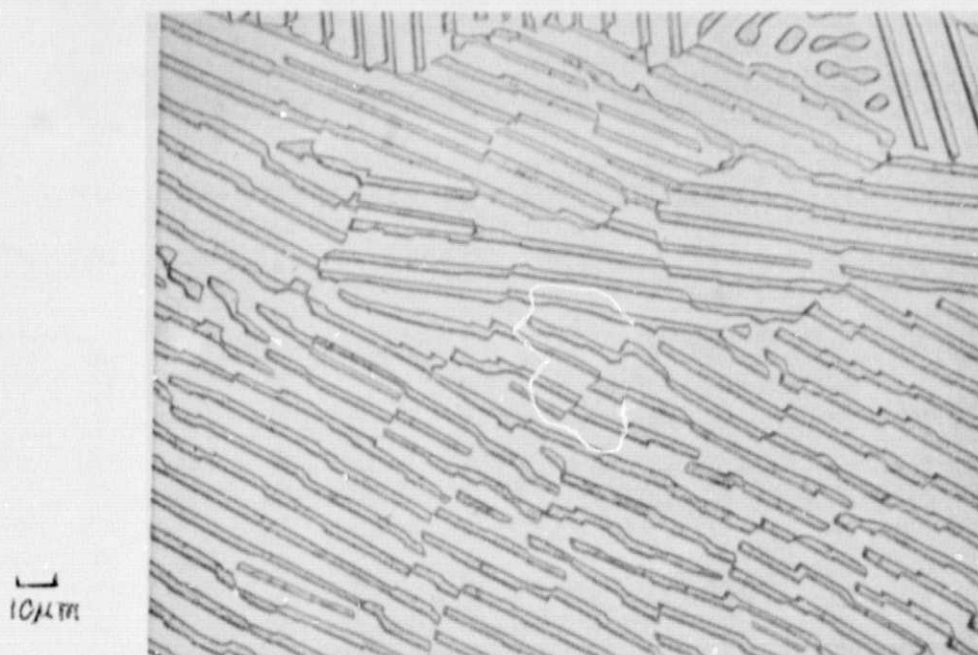[g] Number of fields measured directly on specimen by instrument

Figure 1. Microstructure typical of conventionally cast
superalloys, showing gamma-prime-eutectic
constituent, Specimen L646A. Light micrograph,
original magnification 250X.

a. Rod eutectic (HfC in Co-base matrix), Specimen KIM#80.
    SEM micrograph, original magnification 1300X.

b. Lamellar eutectic ((Fe,Ni)Al lamellae alternating with
    gamma solid solution lamellae), Specimen SNT-9D. Light
    micrograph, original magnification 500X.

Figure 2. Typical microstructures of the two directionally
           solidified (DS) eutectic alloys.
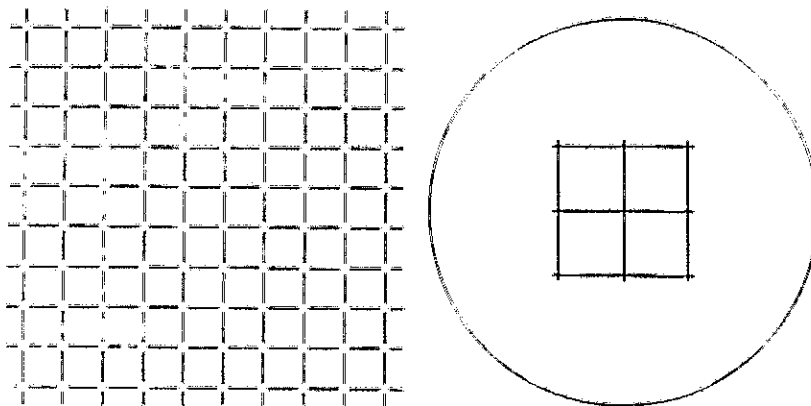
Figure 3. Left: Copy of 100-point macro grid used for manual point
           counting of photomicrographs. Right: Sketch of 9-point
           eyepiece grid used for manual point counting of specimen
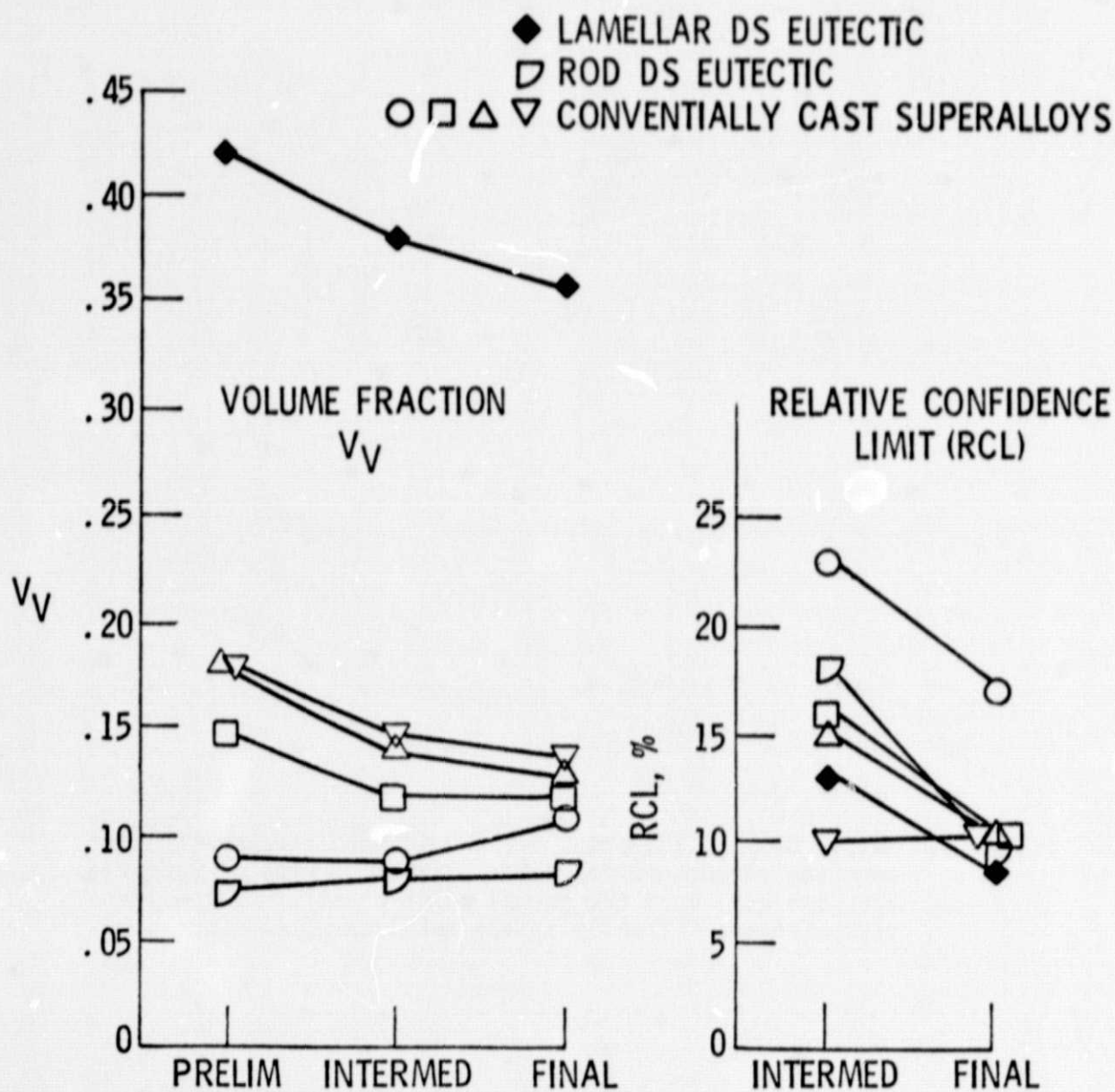   microstructure directly in the metallographic microscope.

Figure 4. Effect of amount of manual point counting done on values of volume fraction (V_V) and relative confidence limit (RCL) obtained.