

METALLOGRAPHIC EVALUATION OF GRAIN SIZE
IN WROUGHT NICKEL AND HEAT RESISTANT ALLOYS

1. SCOPE:

1.1 Purpose: This specification covers the requirements for the evaluation of grain size in bars, forgings, rings, and plate.

1.2 Application: For product having either predominantly uniform or non-uniform grain size, or mixtures of each.

1.3 Classification: This specification covers the following classes;

CLASS A - Usable thru ASTM Grain Size 10

CLASS B - Usable thru ASTM Grain Size 13

2. APPLICABLE DOCUMENTS: The following publications form a part of this specification to the extent specified herein. The latest issue of Aerospace Material Specifications shall apply. The applicable issue of other documents shall be as specified in AMS 2350.

2.1 SAE Publications: Available from SAE, 400 Commonwealth Drive, Warrendale, PA 15096.

2.1.1 Aerospace Material Specifications:

AMS 2350 - Standards and Test Methods

2.2 ASTM Publications: Available from American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

ASTM E112 - Determining Average Grain Size

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3. TECHNICAL REQUIREMENTS:

3.1 Specimen Selection and Preparation: Specimens used for the determination of grain size shall be selected, prepared, etched, and examined at not lower than 100X magnification in accordance with a plan agreed upon by purchaser and vendor.

3.2 Fully Recrystallized or Fully Unrecrystallized Structures: If the grains are fully recrystallized or fully unrecrystallized, the grain size shall be estimated by comparison of a polished and etched specimen with wall comparison charts of transparencies referenced in ASTM E112. When grains are elongated or distorted enough to make the comparison method unsuitable the referee method in 3.6 shall be used. The estimated average ASTM grain size number shall be reported. In addition, estimate the area of the largest grain in the field of view and convert it to an equivalent ASTM grain size number using the tables in ASTM E112, Micro Grain Size-Area of Average Grain Size Relationship. The area and equivalent grain size shall be reported.

3.3 Duplex Structure: When it is determined that a specimen has both recrystallized and unrecrystallized grains, microstructure shall be evaluated by the following procedure; in case of dispute the referee procedure shall be in accordance with 3.6:

3.3.1 Estimate the average ASTM grain size number of the recrystallized grain areas of the specimen (comparison method) as follows:

3.3.1.1 CLASS A: One area, selected at random, shall be evaluated. Grain structure finer than ASTM No. 10 shall be reported as ASTM No. 10.

3.3.1.2 CLASS B:

3.3.1.2.1 If it is determined that the recrystallized grain size is finer than the unrecrystallized grain size, three areas, selected at random, shall be evaluated. The average recrystallized grain size shall be reported. Grain structure finer than ASTM No. 13 shall be reported as ASTM No. 13.

3.3.1.2.2 If it is determined that the recrystallized grain size is coarser than the unrecrystallized grain size, one area, selected at random, shall be evaluated. Grain structure finer than ASTM No. 10 shall be reported as ASTM No. 10.

3.3.2 Estimate the average unrecrystallized ASTM grain size number (comparison method) as follows:

3.3.2.1 CLASS A: One area, selected at random, shall be evaluated.

3.3.2.2 CLASS B:

3.3.2.2.1 If it is determined that the unrecrystallized grain size is finer than the recrystallized grain size, three areas, selected at random shall be evaluated. The average unrecrystallized grain size shall be reported. Grain structure finer than ASTM NO. 13 shall be reported as ASTM No. 13.

3.3.2.2.2 If it is determined that the unrecrystallized grain size is coarser than the recrystallized grain size, one area, selected at random, shall be evaluated. Grain structure finer than ASTM No. 10 shall be reported as ASTM No. 10.

3.3.3 Estimate the percentage of area occupied by recrystallized grains in the specimen being examined (See 3.5).

3.3.4 Determine total average grain size using the Total Grain Size Formula. This formula provides a means to calculate the total average grain size of duplex microstructures.

$$\text{TOTAL AVERAGE GRAIN SIZE} = (-6.6438) + 3.3219 \text{ Log} \left[(X) 10 \left[\frac{\text{GS Rx}}{3.3219} \right] + (100-X) 10 \left[\frac{\text{GS UN-Rx}}{3.3219} \right] \right]$$

WHERE X = Percentage of recrystallized grains
 GS Rx = AVG. GS of recrystallized grains
 GS UN-Rx = AVG. GS of un-recrystallized grains

3.3.5 Alternate Method: Determine the total average grain size graphically using the TAGS parameters.

3.3.5.1 Read the TAGS parameter of the recrystallized structure from the graph presented in Fig. 3 by selecting the intersection of the appropriate recrystallized grain size curve with the percentage of area occupied by the recrystallized grains. The appropriate recrystallized grain size curve shall have a grain size value equal to the average recrystallized grain size (3.3.1).

3.3.5.2 Read the TAGS parameter of the unrecrystallized structure from the graph presented in Fig. 3 by selecting the intersection of the appropriate unrecrystallized grain size curve with the percentage of area occupied by the unrecrystallized grains. The appropriate unrecrystallized grain size curve shall have a grain size value equal to the average unrecrystallized grain size (3.3.2).

3.3.5.3 Add the recrystallized TAGS parameter to the unrecrystallized TAGS parameter.

3.3.5.4 Obtain total average grain size from the graph presented in Fig. 4 by using the summation of TAGS parameters obtained in 3.3.5.3.

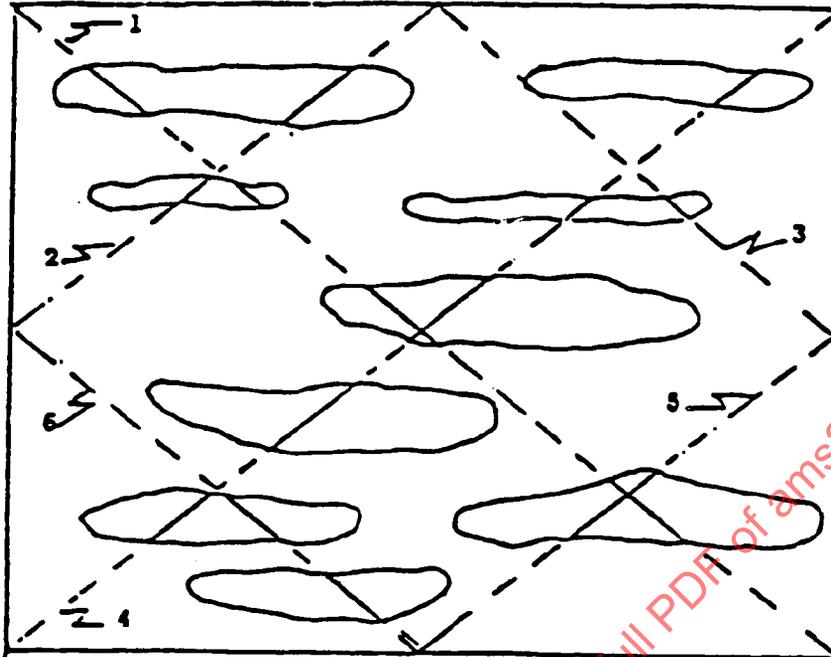
3.3.6 Report the above results in the test report.

- 3.4 Largest Grain: Determine the size of the largest grain by measuring the diameter. When grains are elongated or distorted enough to make this method unsuitable, the area of the grain shall be measured. The diameter or area shall be converted to an ASTM grain size number using the Table of micro-grain size relationships in ASTM E112.
- 3.5 Recrystallized Grains: The percentage of area occupied by recrystallized grains in a specimen shall be estimated by comparison with Figs. 5 to 15.
- 3.5.1 Estimates between 7.5 and 100% shall be rounded to the nearest multiple of 10%.
- 3.5.2 Estimates between 2.5 and 7.5% shall be reported as 5%.
- 3.5.3 Estimates less than 2.5% shall be reported as 0%.
- 3.5.4 In case of dispute, the referee procedure shall be in accordance with 3.6.
- 3.6 Referee Procedures: In case of dispute the following referee procedure shall be used:
- 3.6.1 To establish if grains are unrecrystallized, a sample shall be solution heat treated at least 50°F (30°C) above the original solution heat treat temperature; if grains revert to a smaller size, the sample shall be considered unrecrystallized.
- 3.6.2 To establish average grain size, the Heyn intercept procedure in accordance with ASTM E112 using test lines as shown in Fig. 2 shall be used at the following magnification.
- | | |
|---------|------|
| CLASS A | 100X |
| CLASS B | 250X |
- 3.6.3 To establish the percentage of area occupied by unrecrystallized grains:
- 3.6.3.1 Construct a transparent scale with permanent diagonal lines as in Fig. 1.
- 3.6.3.2 Measure at 100X magnification the lengths of those portions of Line 1 on the scale which pass within the unrecrystallized grains in the specimen as shown in Fig. 1.
- 3.6.3.3 Repeat for lines 2 through 6, inclusive.
- 3.6.3.4 Total the lengths of the scale lines passing within all the unrecrystallized grains on the scale and divide by the total full length of scale lines 1 through 6, inclusive.
- 3.6.3.5 The result multiplied by 100 is the percentage of the area examined which contains unrecrystallized grains.

4. QUALITY ASSURANCE PROVISIONS:

- 4.1 Responsibility for Inspection: The vendor of the product shall supply all samples for vendor's tests and shall be responsible for performing all required tests. Results of such tests shall be reported to the purchaser as required by 4.4. Purchaser reserves the right to sample and to perform any confirmatory testing deemed necessary to ensure that the product conforms to the requirements of this specification and the material specification.
- 4.2 Classification of Tests: Tests to determine conformance to all technical requirements of this specification are classified as acceptance tests and shall be performed on each heat or lot as applicable.
- 4.3 Sampling: Shall be as agreed upon by purchaser and vendor.
- 4.4 Reports: The numerical results of all evaluations performed in accordance with 3.2 through 3.6 shall be reported in the certificate of test accompanying the material in addition to other information required by the applicable material specification.
- 4.5 Resampling and Retesting: If any specimen used in the above tests fails to meet the specified requirements, disposition of the product may be based on the results of testing three additional specimens for each original nonconforming specimen. Failure of any retest specimen to meet the specified requirements shall be cause for rejection of the product represented and no additional testing shall be permitted. Results of all tests shall be reported.
5. PREPARATION FOR DELIVERY: Not applicable.
6. ACKNOWLEDGMENT: A vendor shall mention this specification number in all quotations and when acknowledging purchase orders.
7. REJECTIONS: Not applicable.
8. NOTES:
- 8.1 Inspection procedures meeting the requirements of this specification have been classified under Federal Standardization Area Symbol "NDTI"

This specification is under the joint jurisdiction of AMS Committees "B" and "F".

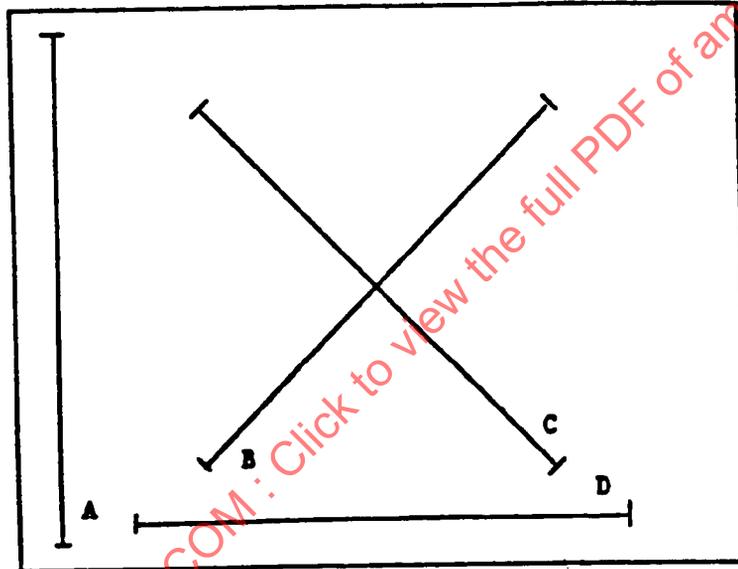


Line Number	Line Length Inches (mm)	Unrecrystallized Line Length-Inches (mm)				
1	5.70 (142.5)	0.40 (10.0)	0.15 (3.8)	0.50 (12.5)	0.60 (15.0)	
2	2.85 (71.2)	0.20 (5.0)	0.50 (12.5)			
3	2.85 (71.2)	0.10 (2.5)	0.20 (5.0)			
4	5.70 (142.5)	0.45 (11.2)	0.65 (16.2)	0.60 (15.0)	0.20 (5.0)	0.35 (8.8)
5	2.85 (71.2)	0.60 (15.0)				
6	2.85 (71.2)	0.40 (10.0)	0.40 (10.0)			
Total	22.80 (569.8)			6.30 (157.5)		

$$\text{Unrecrystallized Grain Area} = \frac{6.30 \text{ in. (157.5 mm)}}{22.80 \text{ in. (569.8 mm)}} = 27.5\%$$

$$\text{Recrystallized Grain Area} = 100 - 27.5\% = 72.5\%$$

Figure 1 - Example of Intercept Method of Measuring Unrecrystallized Grain Area



Total Line Length = 275 mm min
Individual Line Length = Total Line Length/4
Angles BC and AD = 90°
Angle AB = 45°
Only Lines B and C Intersect

Figure 2 - Example of Hyen Intercept Test Lines

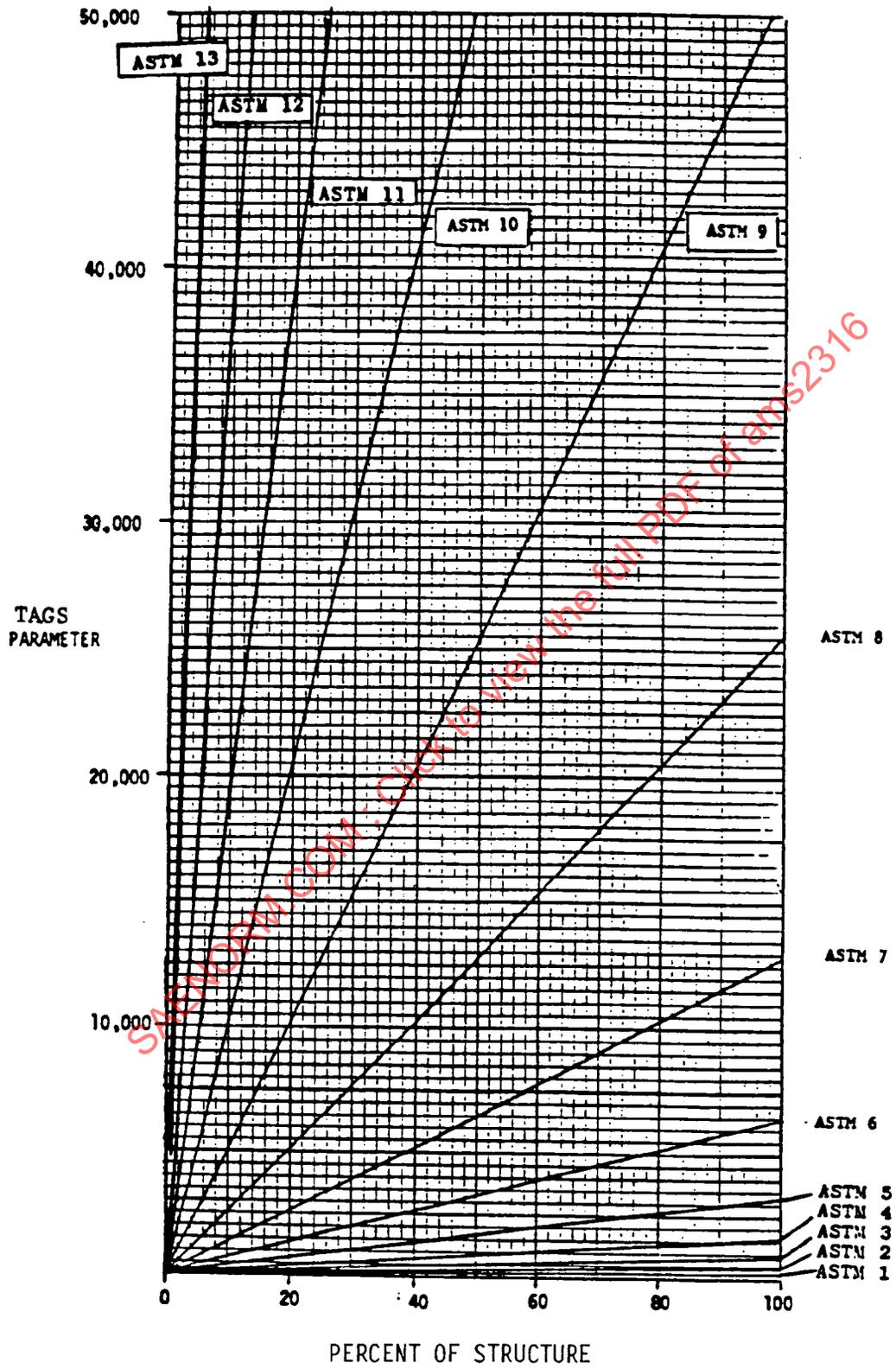


Figure 3 - TAGS PARAMETER

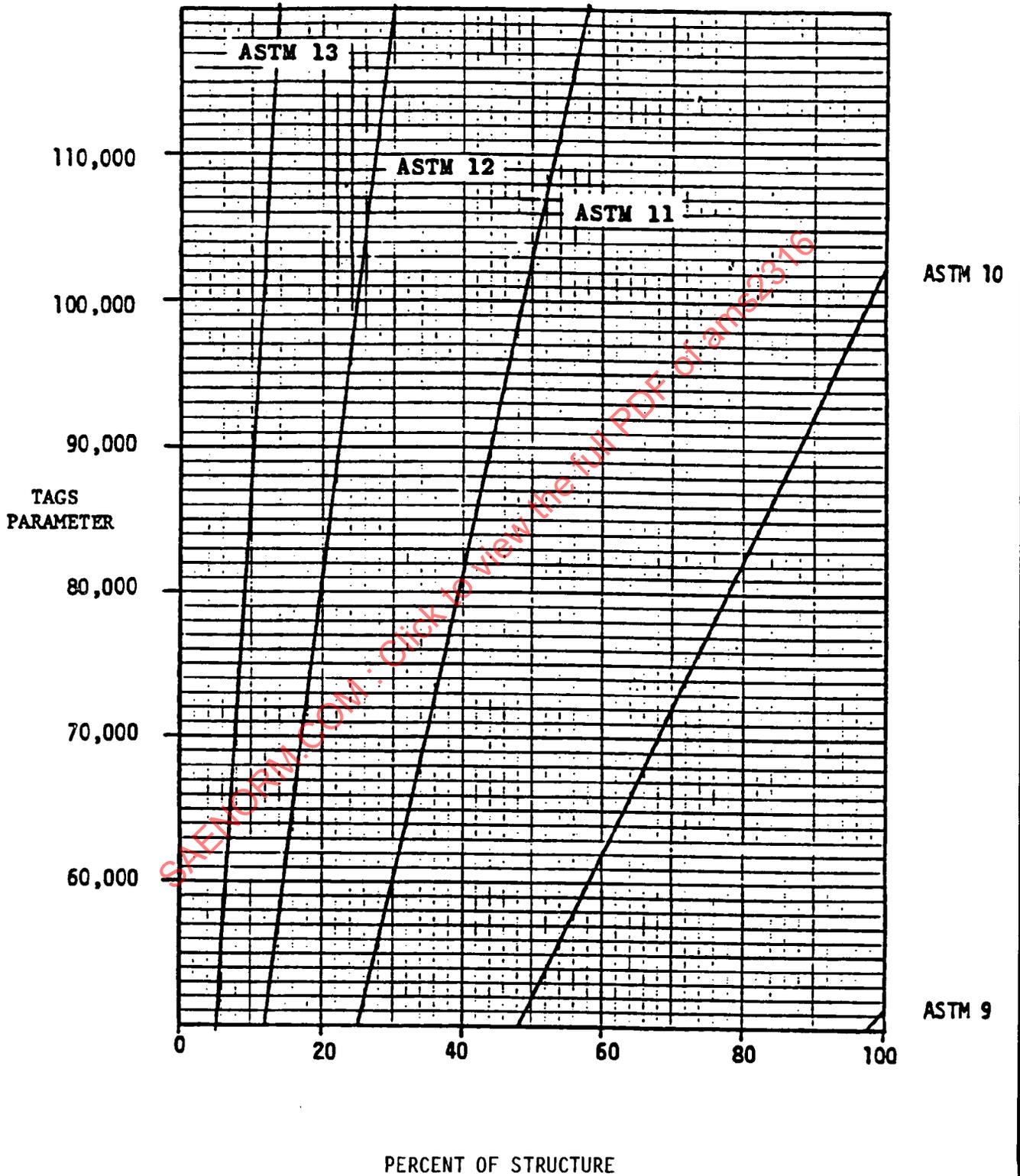
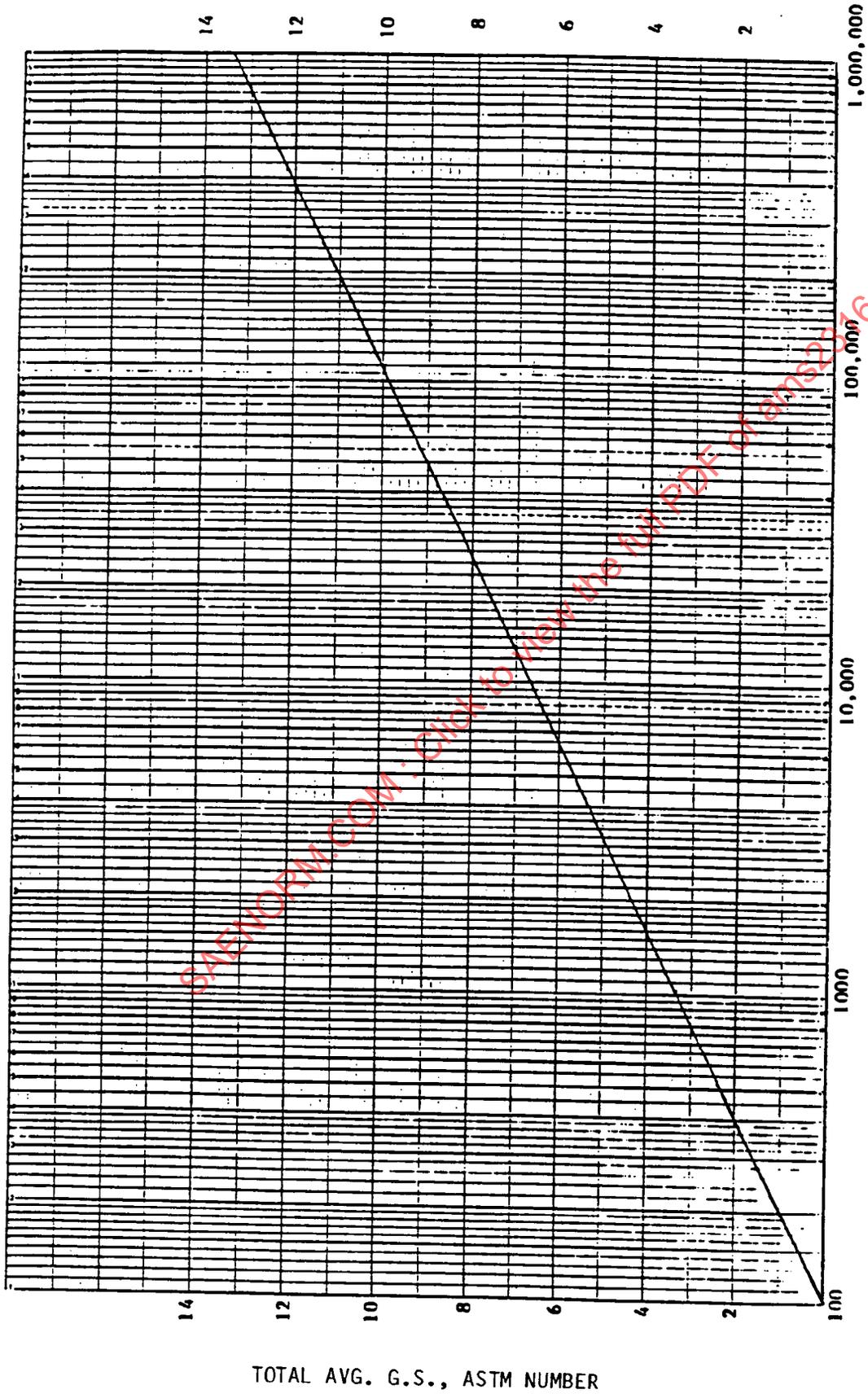
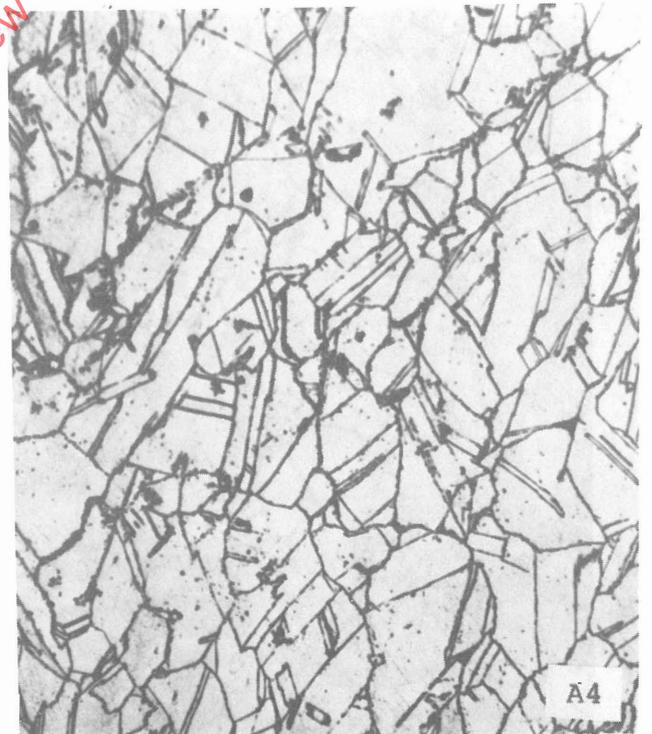
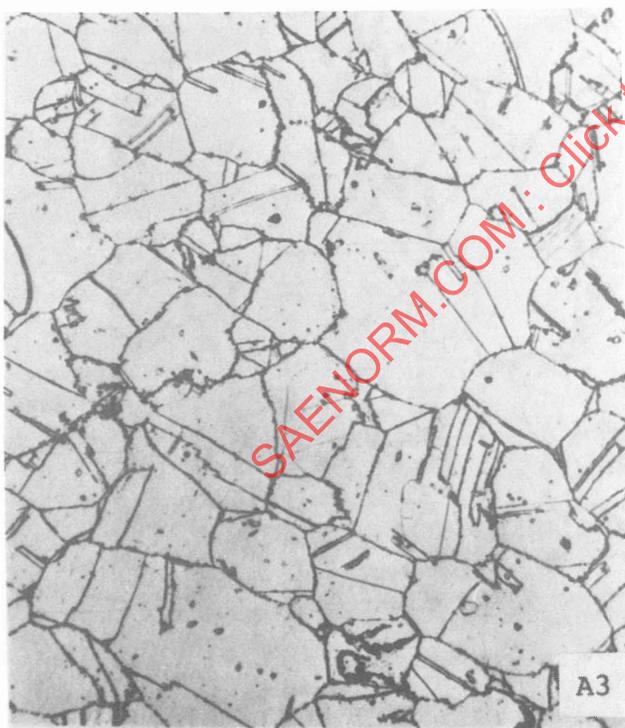
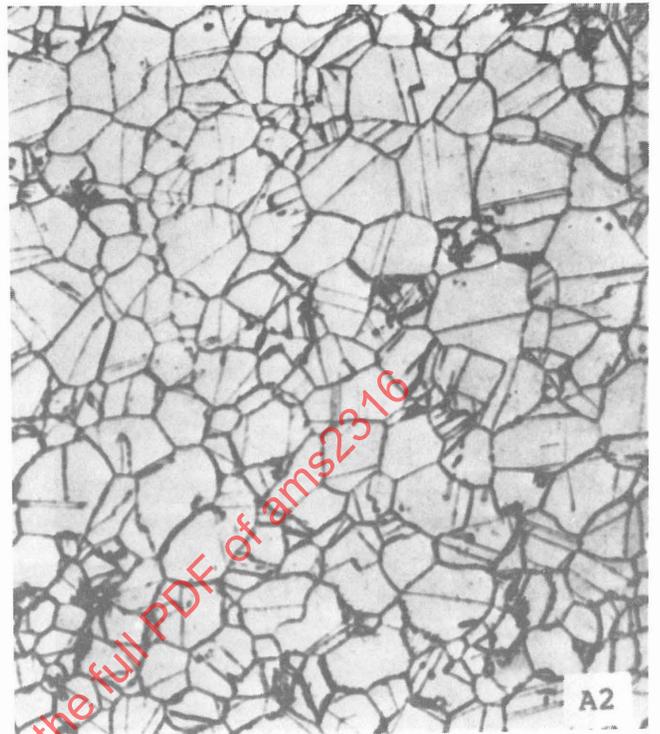
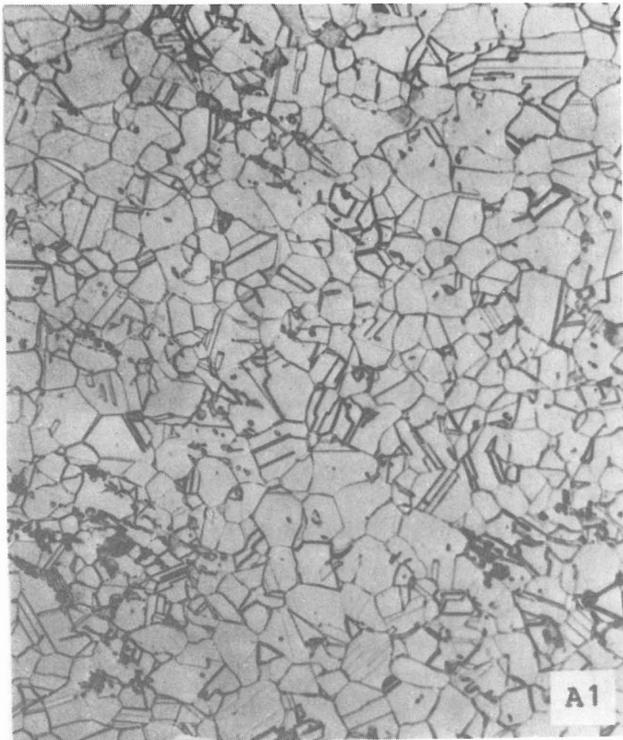


Figure 3 - TAGS PARAMETER (Continued)

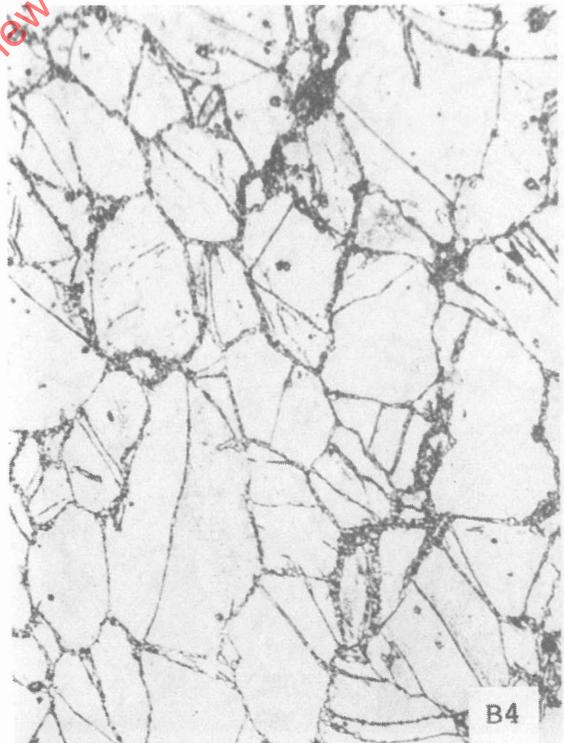
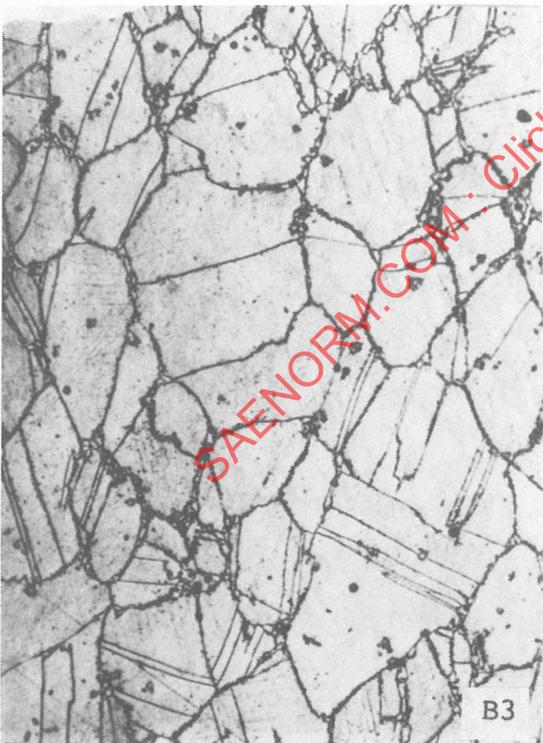
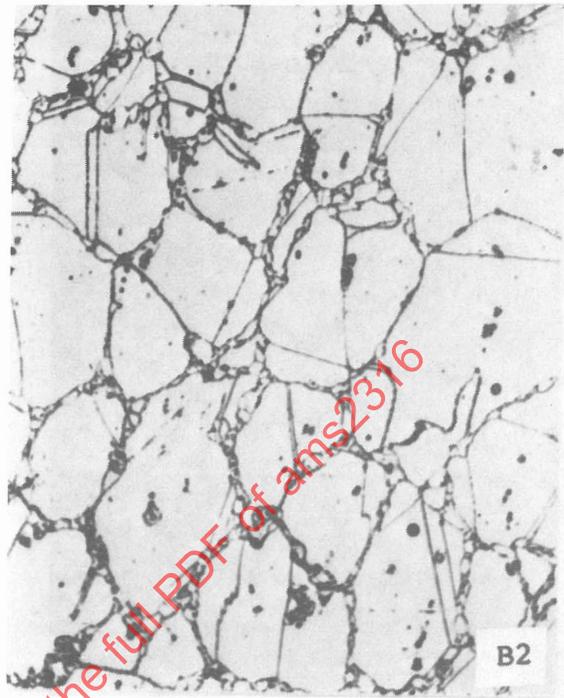
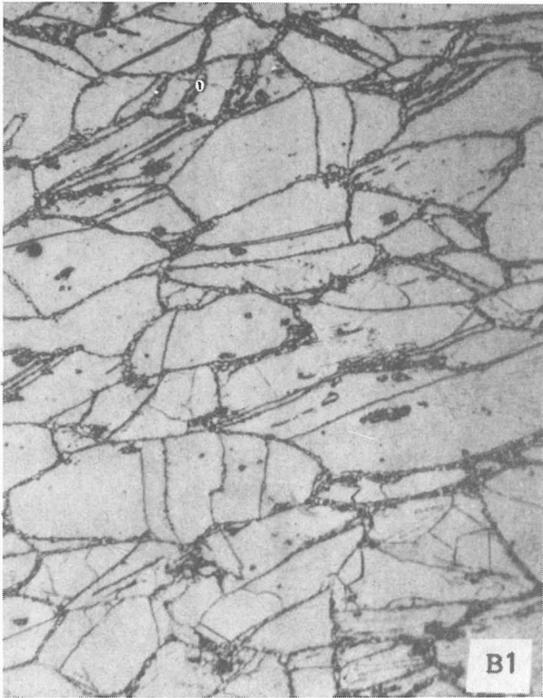


TAGS PARAMETERS SUMMED
FIGURE 4 TOTAL AVERAGE



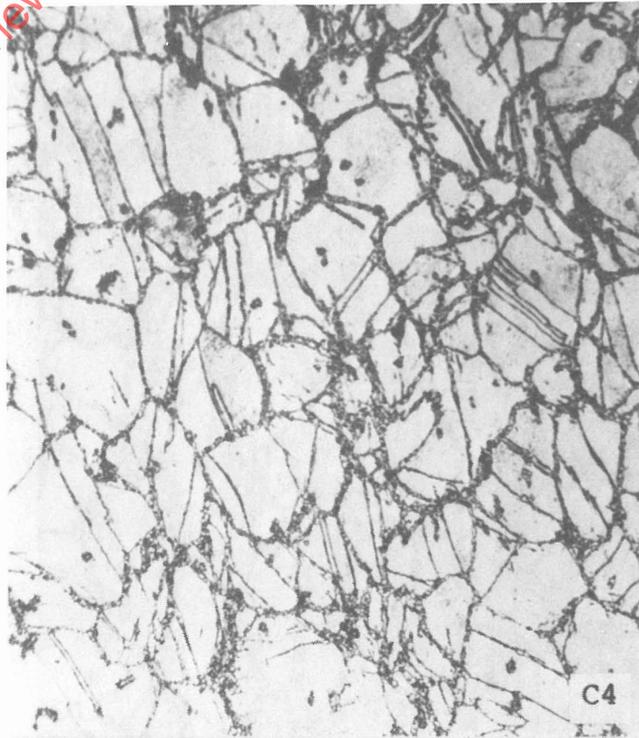
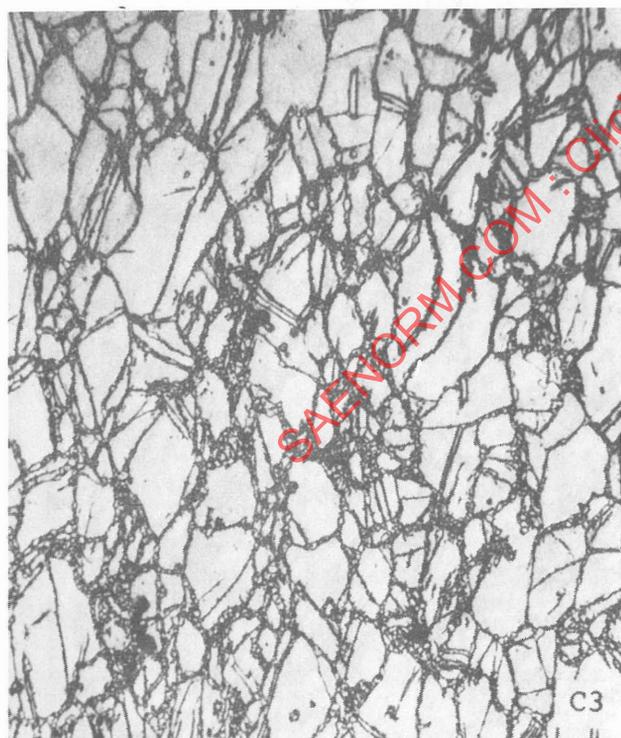
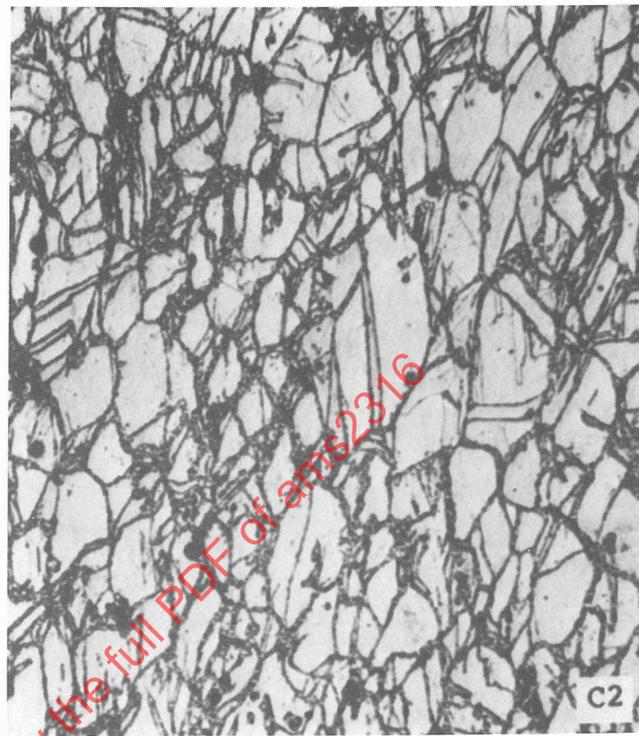
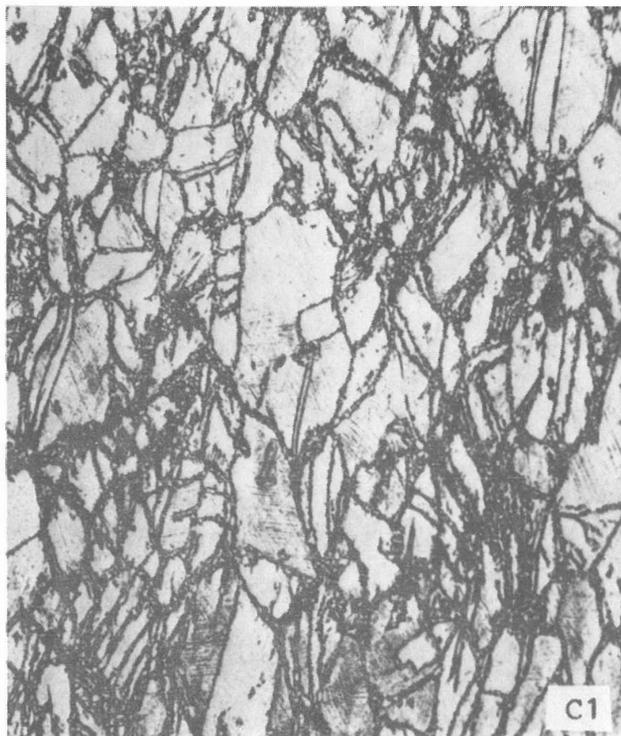
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Figure 5



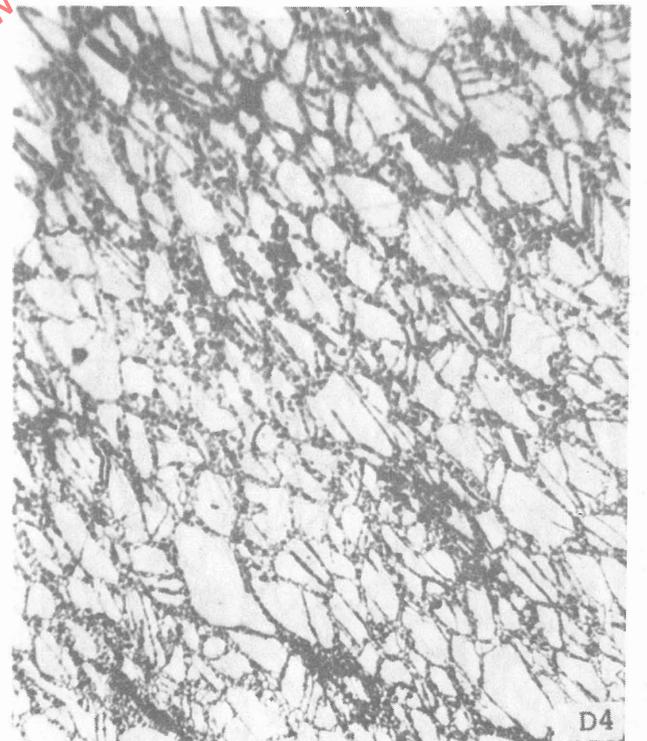
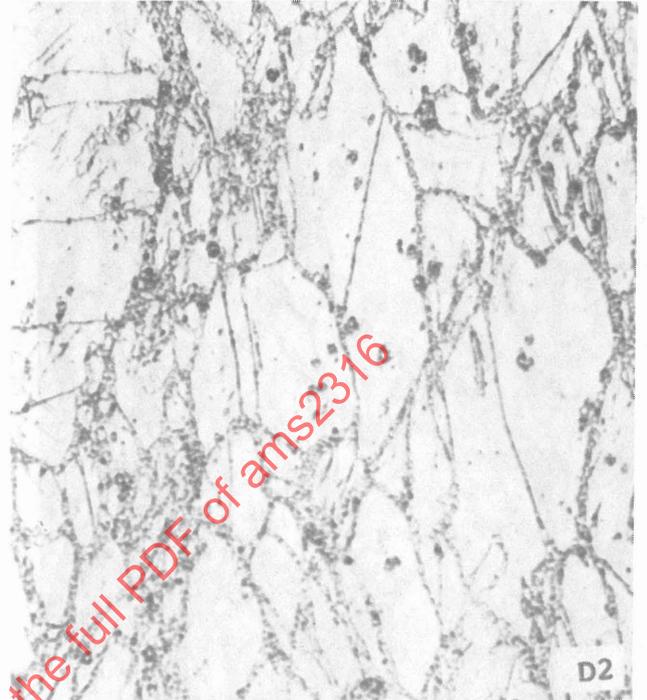
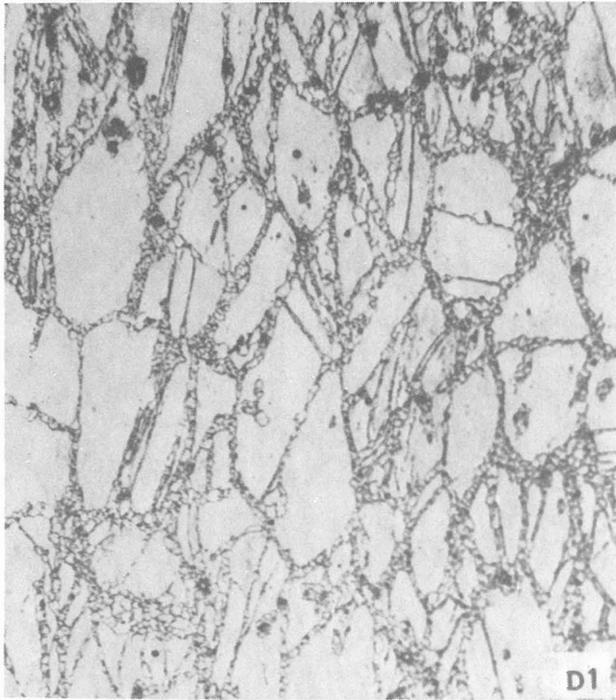
10 PERCENT RECRYSTALLIZED

Figure 6



20 PERCENT RECRYSTALLIZED

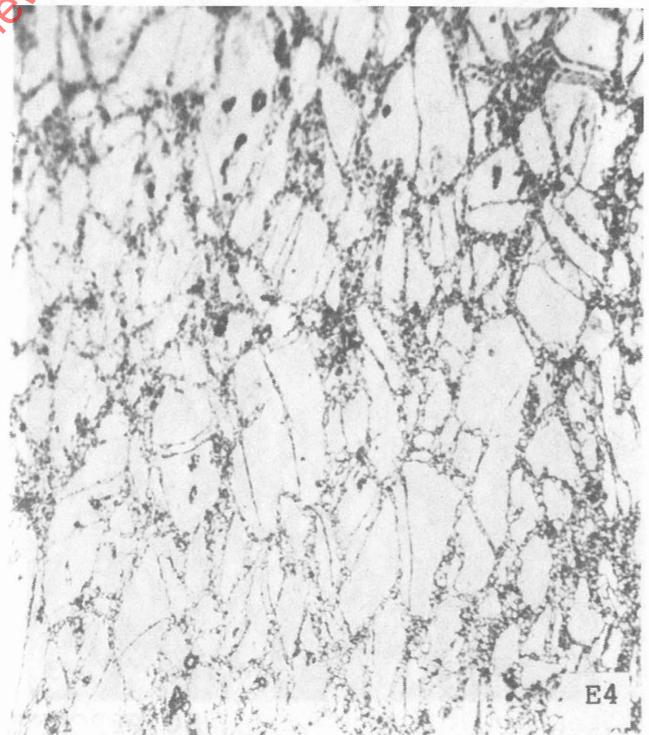
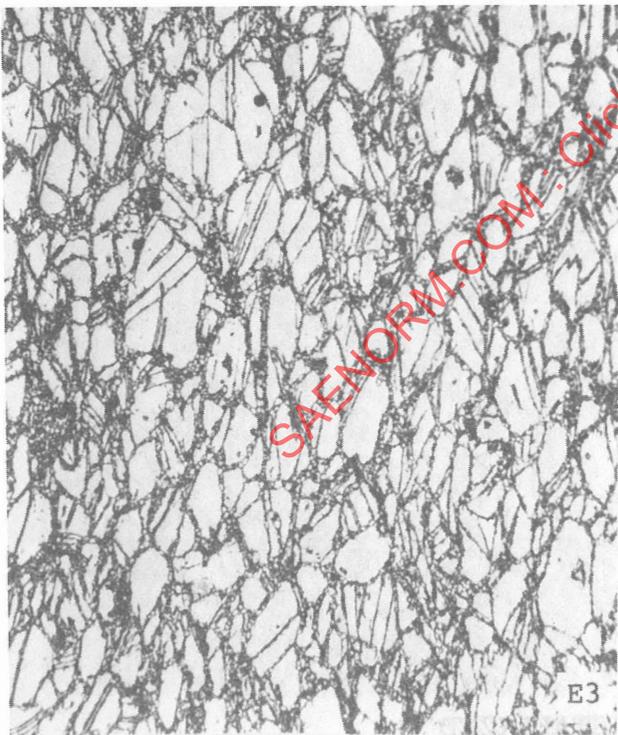
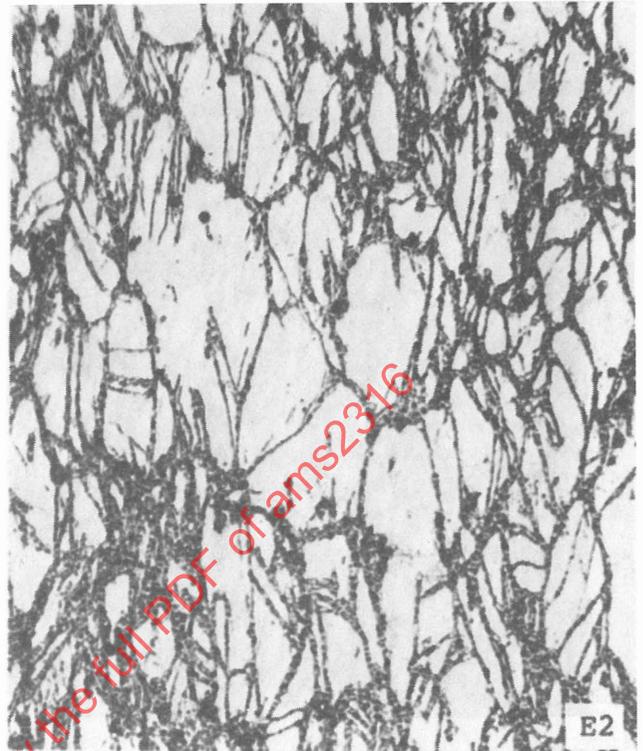
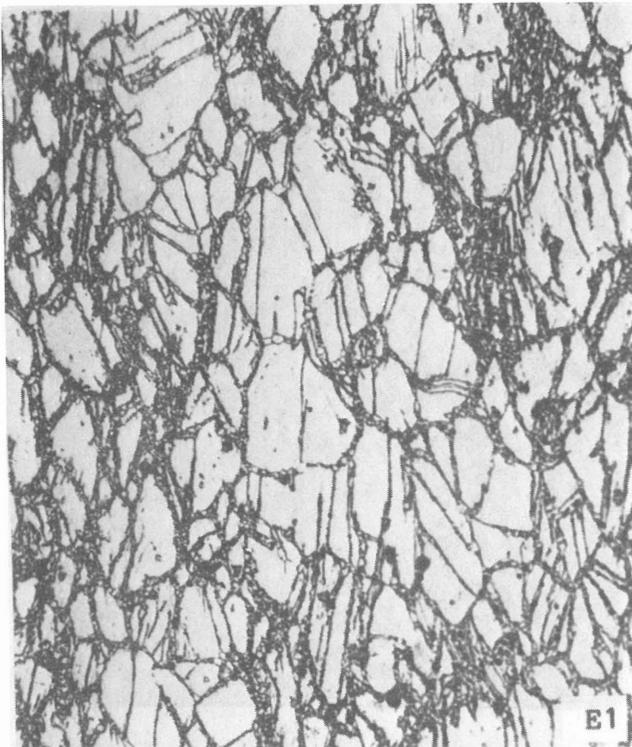
Figure 7



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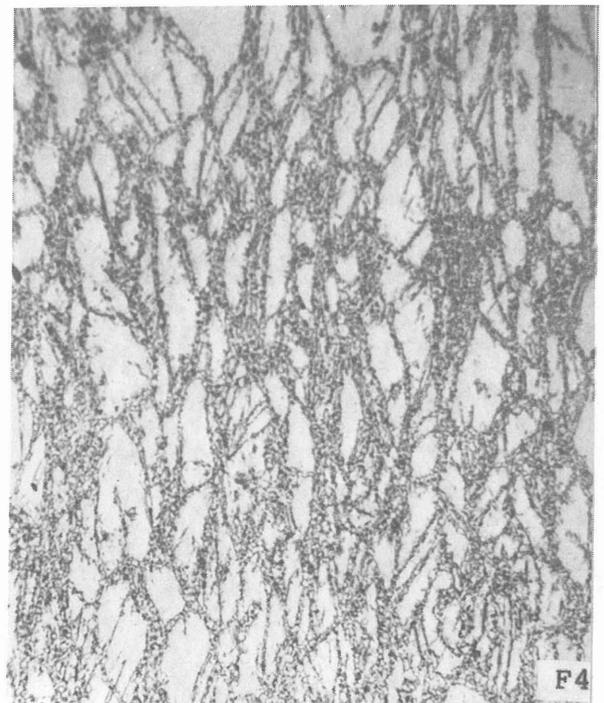
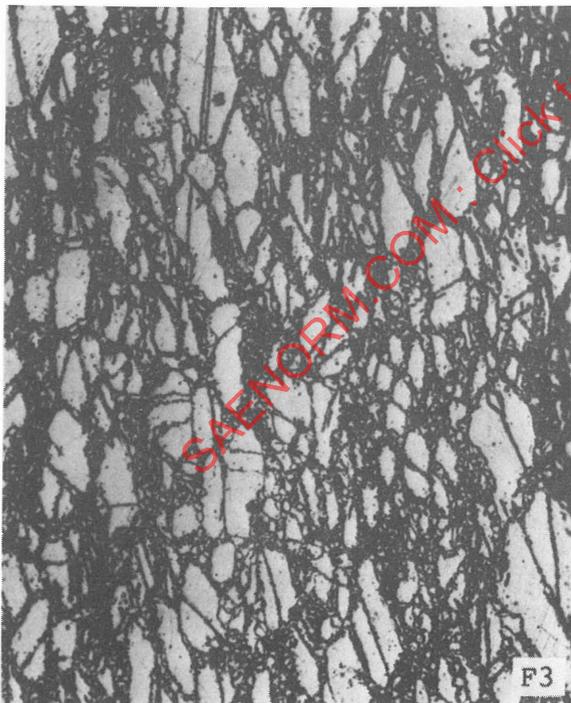
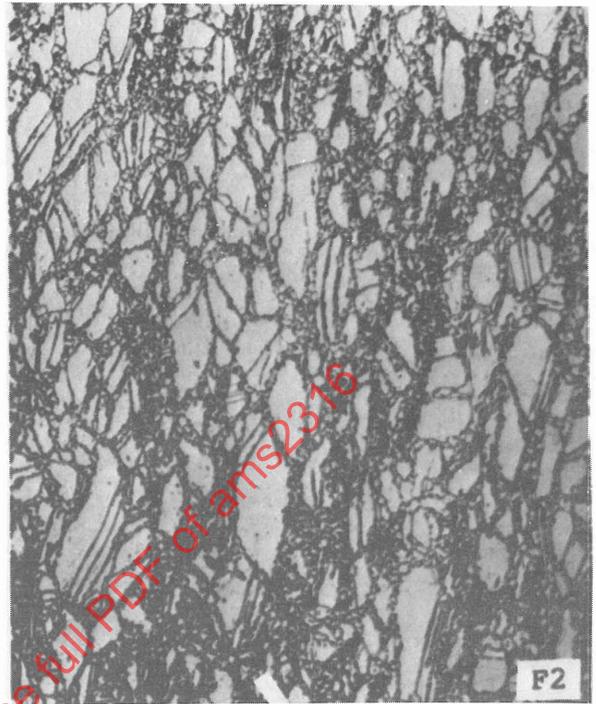
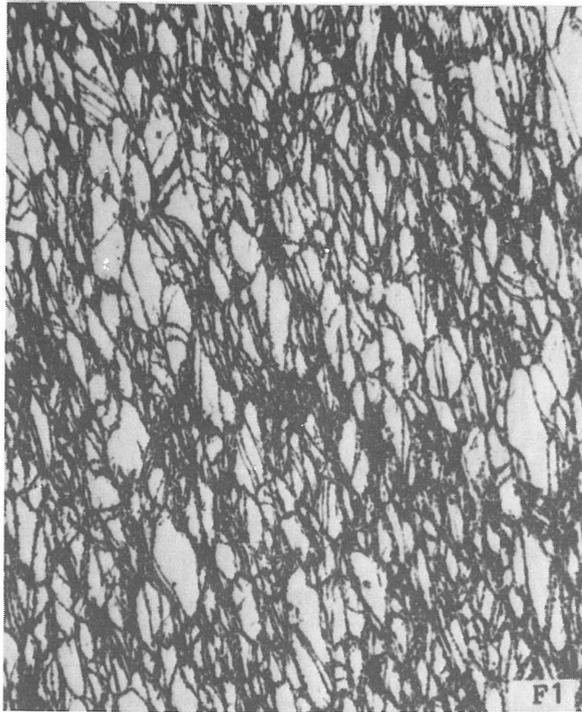
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Figure 8



40 PERCENT RECRYSTALLIZED

Figure 9



50 PERCENT RECRYSTALLIZED

Figure 10