CONTROLLERATE OF QUALITY ASSURANCE(METALS) ICHAPUR

QUALITY ASSURANCE INSTRUCTIONS

ON

STEELS FOR BARREL EXTENSION, BREECH BLOCK AND PISTON EXTENSION

QAI NO. QA-1/QAI/15

1. SCOPE:

1.1 This Quality Assurance Instructions (QA-1/QAI/15) are made to govern supply and quality assurance of steel bars to be used for highly stressed / critical components of Small Arms, namely Barrel Extension (DS Cat No. 1005-007854), Breech Block (DS Cat No. 1005-007778) and Piston Extension (DS Cat No. 1005-007792). These instructions are in addition to existing specification, BS-970-Pt-I, 83.

2. **RELATED SPECIFICATION**

a. BS: 970 Pt-I – 1983

b. IS: 4163-2004

c. BS:4490

d. IS: 13015 – 1992

e. ASTM E 112

f. ASTM E 381

g. IS: 228

h. IS: 1757

i. IS: 1598

i. IS: 1608

3. MATERIAL

- 3.1 The bars shall be supplied peeled, straight and free from any type of surface defects.
- 3.2 The Chemical composition of the steel to be checked as per IS: 228 (Pt-1-6 & 8-10) shall conform to the requirements of relevant grades to BS:970 (Pt-I)- 83 as under:

Component	Speen.	C%	Mn%	Cr%	Mo%	Ni%	S%	P%	S+P%	Si%
Barrel Extension	BS-970 Pt-I 83 Gd 817 M40 (T)	0.36 0.44			0.20 0.35			0.015 max)		0.10 0.35
	(Table-6)									

Component	Specn.	C%	Mn%	Cr%	Ni%	S%	P%	S+P%	Si%
Piston Extension	BS-970 Pt-I 83 Gd 655 M13,	0.10 0.16	0.35 0.60	0.70 1.00	3.0 3.75	0.010 (max)	0.015 max)	0.022 (max)	0.10 0.35
	Table-11								

Component	Specn.	C%	Mn%	Cr%	Mo%	Ni%	S%	P%	S+P%	Si%
Breech	BS-970 Pt-I 83	0.12	0.25	1.00	0.15	3.90	0.010	0.015	0.022	0.10
Block	Gd 835 M15	0.18	0.50	1.40	0.30	4.30	(max)	max)	(max)	0.35
	(EN39B),									
	Table-11									

3.3 Deviation permitted in chemical composition

The deviation permissible in chemical composition as per BS:970 Pt-I (Table-1) is as under:

Barrel Extension		Piston	Extension	Breech Block		
C%	± 0.02	C%	± 0.01	C%	± 0.01	
Mn%	± 0.03	Mn%	± 0.03	Mn%	± 0.03	
Cr%	± 0.04	Cr%	± 0.04	Cr%	± 0.04	
Ni%	± 0.05	Ni%		Ni%	± 0.07	
Mo%	± 0.02	Mo%	5 -	Mo%	± 0.02	

4. **MANUFACTURE**

- 4.1 The Steel shall be produced in basic electric process followed by VD. Steel should be free from surface defects like cracks, piping, flaws and ingotism.
- 4.2 Austenite grain size of the steel when determined in accordance with IS:4748, latest edition shall be in the range of 5-8.

NOTE

Grain size of the steel shall be determined when specifically called for in order / contract and agreed to between the purchaser and supplier.

5. MARKING

- 5.1 All Bars shall legibly and indelibly be marked carried out with the proper identification. The identification on the bars shall be maintained till final acceptance of the steel:
 - (a) Cast No. / Batch No.
 - (b) Code of reference
 - (c) Manufacturers logo & Year of Manufacture.

6. PRE-INSPECTION OF THE STORES/ CONSIGNMENT

6.1 Manufacturers / Suppliers must satisfy themselves that the stores are in accordance with their terms of contract and fully conformed to the required specifications by carrying out a thorough pre-inspection of each lot before actually tendering the same for inspection to Inspection Authority nominated under the terms of contract. Deceleration by the manufacturer / supplier that necessary pre-inspection has been carried out on the stores tender will be submitted along with the challan / material TCs

6.2 If the Inspecting Authority finds that the pre-inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

7. SAMPLING / SELECTION OF TEST SAMPLES

7.1 Bars supplied as normalized, normalized and tempered, annealed or hardened and tempered condition

- 7.1.1 The test piece shall be cut longitudinally so that its axis is 12mm below the surface of the test sample, the nearest end of the test piece being 300mm or more from end of the rolled length.
- 7.1.2 For every 100 bars or parts thereof the same cast and heat-treatment batch, one test sample shall be selected for test(s) in the same manner as per rolled bar but the test pieces may be taken from the end of the bar.
- 7.2 Where Impact Test is specified, the test pieces shall be taken from the equivalent positions to those of tensile test pieces and shall be subjected to identical heat-treatment.
- 7.3.1 The details of actual heat-treatment shall be recorded on test certificate and or released certificate.

8. QUALITY CONTROL

- Quality control of the material shall be carried out by the Quality Control Manager of the Firm by assuring that the inspection of material tendered has been carried out as per procedure given in Appendix 'A' (for Bloom, Billets & Slabs), and Appendix 'B' (for rolled Bars).
- 8.2 Testing as stipulated in clause 8.3 shall be carried out by the Quality Control Manager of manufacturer.

8.3 TESTING

8.3.1 Mechanical Test; Tensile Test including Yield Stress determination shall be done in accordance with IS:1608. The Impact Test shall be carried out in accordance with IS:1757 and IS:1598. The result of the test should satisfy all the requirements of relevant grade of specified material to BS:970-83 Pt-I as under

8.3.2 MECHANICAL PROPERTIES

Component	UTS	YS	%E	Impact	Hard	ness
Barrel	MPa	MPa	(5.65√So)	Izod	'T'	Annealed
Extension				Ft lbf	Condition	
BS:970 Pt.I	850	680	13	40	248-302	277 HB
83 Gd 817	1000	Min	Min	Min	HB	(max)
M40 'T'						

Component	UTS	YS	%E	Impact	Haro	lness	Case
Breech	MPa	MPa	(5.65√So)	Izod	Case	Annealed	depth
Block				Ft lbf	Hardness		in
							mm
BS:970	1300	-	8	25	575-650	-	0.13
Pt.I 83 Gd	min.		min.	min.	HB		-
835 M15							0.26
(EN 39B)							

Component	UTS	YS	%E	Impact	Haro	lness	Case
Piston	MPa	MPa	(5.65√So)	Izod	Case	Annealed	depth
Extension				Ft lbf	Hardness		in mm
BS:970	1000	-	9	30	500-575	-	0.13
Pt.I 83 Gd	min.		min	min.	HB		-
655 M13							0.26

9. <u>DETERMINATION OF INCLUSION CONTENT BY</u> <u>MICROSCOPIC METHOD</u>

9.1 Inclusion content for sizes, shapes and type of inclusion shall be carried out in accordance with IS:4163-2004 and the following acceptance standards shall be followed:

Series	Sulfide (A)	Aluminate (B)	Silicate (C)	Globular Oxide (D)	Single Globular (DS)
Thin (Max)	1.5	1.5	1.5	1.5	
Thick(Max)	0.5	Nil	Nil	0.5	1.0

10. MICRO-EXAMINATION

- 10.1 The micro examination of the steel shall also be carried out by taking samples from the finished size of the material to ensure freedom from Slag inclusions, Rolled-in-scale etc.
- 10.2 Qualitative assessment of Banding shall be done in annealed condition of the input material of Barrel extension. Banding of ferrite pearlite structure should comply to Series 'C', Point 1, Scale-3 of Table-3 to GOST 5640-68.
- 10.3 Qualitative assessment of Banding shall be done in annealed condition of the input material of Piston Extension. Banding of ferrite pearlite structure should comply to Series 'A', Point 1, Scale-3 of Table-3 to GOST 5640-68.
- 10.4 Qualitative assessment of Banding shall be done in spherodized annealed condition of the input material of Breech Block. The micro structure shall be uniformly dispersed globular carbide.
- 10.5 The microstructure of the hardened & tempered steel shall be uniformly tempered martensite in case of Barrel Extension.
- 10.6 Micro-structure of case of the case carburised Breech Block and Piston Extension shall be tempered martensite with free from massive / network carbides.

11. MACRO-ETCH TEST OF CLEANLINESS OF STEEL

- 11.1 Full cross-section samples shall be taken after discard from billets representing the top and bottom of the first, middle and last usable ingots of each heat and macro-etched as per the procedure laid down in the Appendix 'C'.
- 11.2 The macro structure of the steel for the suitability of small arm components namely Breech block, Barrel extension and Piston extension shall be compared with plates I and II of IS:13015 and conform to the acceptance standards as under:

Nature	Acceptance Standards
Breech block, Barrel extension and Piston extension	Better than or equivalent to C-1, R-1, S-1 for plate-I and NIL for Plate-II.
manufacture from basic electric arc furnace followed by VD	

11.3 The micro examination of the steel shall also be carried out by taking samples from the finished size of the material to ensure freedom from Cracks, Flakes, rolled in scale and slag inclusion etc.

12. MPI TEST

- 12.1 Magnetic Particle flaw detection test (MPI) shall be carried out on 100% bars at supplier end. The details such as name of technician, NDT level-II certificate issued by ASNT / ISNT to the technician and endorsement for conducting 100% MPI test shall be incorporated in relevant TCs by the supplier.
- 12.2 MPI at consignee end shall be done on 10% of bars by certified MPI level-II technician. Any contraindicated bar shall be segregated and tested by way of metallography.
- 12.3 Wet method to clause 7.3(a) of IS 3703 (latest edition) shall be adopted while carrying out the MPI test.
- 12.4 All bars subjected to MPI test should be free from any flaw indication.

13. <u>SELECTION AND TESTING OF AUDIT SAMPLE OF INPUT</u> MATERIAL

On receipt of the Input Material the concerned Ordnance Factories shall intimate to the concerned resident SQAEs along with a copy of MIS and Pre-Inspection material TCs. Subsequently the resident SQAEs / AHSPs shall draw the sample as per sampling plan and conduct the testing in NABL Accredited AHSP / OFs Laboratory for Chemical Composition, Mechanical Properties, Grain Size, Micro Examination & NMIR.

14 RE-TEST

- 14.1 If any one of the sample first selected fails, double number of the sample from the same batch / cast shall be taken further, one of which shall be from the bar from which the original test sample was taken
- 14.2 In case, all the additional samples pass, the batch / cast represented by test samples shall be deemed to comply with the test.
- 14.3 If either of these additional samples fails the batch / cast represented by the test samples shall be rejected.
- 14.4 For material supplied in the heat-treated condition, the steel maker has right to re-heat treat the material under test and re-submit it for inspection and testing. Such heat-treatment shall be limited to twice only.

APPENDIX 'A'

A. INSPECTION PROCEDURE FOR BLOOMS, BILLETS AND SLABS

- A.1 During the process, Inspection and testing the Quality Control Manager is to be satisfied that:-
- A.1.1 All Blooms, Billets and Slabs are produced from ingots which comply with the following requirements: -
- A.1.1.1 The shape, size and method of production of an ingot shall be so selected as to minimize segregation and axial unsoundness. The ingots may be cast wide end up. Specific process for steel making will also be employed when called for in the tender or contract.
- A.1.1.2 The maximum mass of the ingots shall not exceed the specified mass.
- A.1.1.3 The cast analysis should be within the specified limits as stipulated in the order/contract or Table I.

- A.1.1.4 The ingots, unless charged hot into the mill or forge re-heating furnace, should be cooled in a manner which will ensure freedom from any internal or external cracking.
- A.1.1.5 The material on any re-heating and subsequent cooling should not produce harmful defects viz. hairline cracks or clinks.
- A.1.1.6 Where overall dressing is required, the material shall be machined, ground all over at a suitable stage.
- A.1.2 The identity of blooms, billets and slabs according to cast number is maintained through all the stages of processing.
- A.1.3 Re-heating for hot working and cooling after hot working are controlled to prevent the formation of harmful defects viz. hair line cracks or clinks.
- A.1.4 Adequate top and bottom discard have been taken in all the cases to ensure soundness and freedom from piping and harmful segregation.
- A.1.4.1 This is to be proved by Sulphur Print, Macroetch or other methods acceptable to the Quality Control Manager. The test method employed and the positions tested within the blooms, billets or slabs or finished rolled product in case of material rolled directly from ingot to finished product, are to be related to the production method and are to have prior agreement of the Quality Control Manager.
 - (a) Tests on the first and last ingots in a cast comprising 10 ingots or less.
 - (b) Tests on the first, middle and last ingots of a cast comprising more than 10 ingots.
 - (c) One test per plate in the case of an up hill teemed cast
- A.1.5 When called for in the contract/order/specification, samples have been selected in a random manner at a rate of not less than three per cooling batch and flaw detected by a method approved by the Quality Assurance Authority.
- A.1.5.1 If a batch has not cooled in a uniform manner, additional samples are to be selected from that portion of the cast which has cooled more rapidly. Where evidence of hair line cracks is found in test samples, all the material from that cast or cooling batch is to be fully tested by ultrasonic means and all the defective material shall be rejected.

- A.1.6 When called for in the contract/order/specification, the metallographic examination has to be carried out and should show that the steel is of an acceptable level of cleanliness.
- A.1.7 When called for in the contract/order/specification, the sample or samples are to be subjected for the hardenability test and the test results should show satisfactory data as per the requirements.
- A.1.8 If additional discard has been proved necessary by any one of the preceding tests specified in Clauses A.1.4 to A.1.7 above, this discard has been taken and a further similar test has proved the remainder of that ingot product to be satisfactory. In addition, further similar tests are to be taken as follows: -
 - (a) For a direct poured cast the same test to all ingots not tested
 - (b) For up hill poured cast the same test is applicable to all ingots on the same plate as the defective ingots.
 - (c) All additional discard proved necessary by these tests are to be removed.
- A.1.9 When called for in the contract/order/specification, the inherent austenitic grain size as defined in IS 4748 has been determined.
- A.1.10 When called for in the contract/order/specification, check product analysis have been taken and agree reasonably with the cast sample analysis.
 - (a) Such product analysis is to be taken into account for the heterogeneity normal to the steel.
 - (b) Guidance should be obtained on suitable tolerances from the tables showing permitted variations on product analysis from specified range in IS 1570.
- A.1.11 In cases where the contract/order/specification specifies forging quality billets, they are examined visually after suitable preparation and proved free from harmful surface defects. If any of the billet shows harmful surface defects, it shall be rectified by agreement with the purchaser.
- A.1.12 When called for in the contract/order/specification, non destructive testing has been carried out to a technique and a standard of acceptance agreed by Quality Control Manager.

- A.1.13 Any heat-treatment called for in the contract/order/specification has been carried out and the record of temperature comply with the requirement.
- A.1.13.1 Pyrometers of proved accuracy shall be used.
- A.1.14 When called for in the contract/order/specification, blooms, billets and slabs have been machined or dressed as appropriate and the precautions are to be taken to ensure that any hot dressing does not produce harmful defects.
- A.1.15 Any mechanical testing called for in the contract/order/specification has been carried out in accordance with Clause 8.3.1.
- A.1.16 All accepted blooms, billets and slabs have been identified with the Quality Control Manager's stamp and such other marking as will enable the following details to be established.
 - (a) Manufacturer
 - (b) Cast Number
 - (c) Test number where appropriate
 - (d) Specification

APPENDIX 'B'

B. INSPECTION PROCEDURE FOR ROLLED BARS

- B.1 During the process, inspection and testing, the Quality Control Manager is to be satisfied that: -
- B.1.1 All rolled bars are produced from material which complies with the relevant requirements of inspection procedure for Blooms, Billets and Slabs (see Appendix A).
- B.1.2 The identity of bars according to Cast number is maintained through all the stages of processing.
- B.1.3 Re-heating for hot working and subsequent cooling is controlled to avoid deleterious defects.
- B.1.4 Dimensions and straightness of all bars are in accordance with the requirements in the contract/order/specification.
- B. 1.5. All rolled bars are inspected visually and the bars having any visible cracks or other harmful defects are rejected unless the affected portions of the bars or the defects, are satisfactorily removed.
- B.1.5.1 When the contract/order/specification requires that the rolled bars are to be descaled, the visual inspection is to be carried out after the descaling operation.
- B.1.6. When called for in the contract/order/specification non-destructive testing has been carried out to a technique and to a standard of acceptance, agreed by the Quality Control Manager.
- B.1.7. Where the contract/order/specification specifies forging quality steel, it is examined visually after suitable preparation and proved free from harmful surface defects. If any of the steel shows harmful defects, it may be rectified by agreement with the purchaser.
- B.1.8 Any heat-treatment called for in the contract/order/specification has been carried out and the records comply with the requirements. Pyrometers of proved accuracy shall be used.
- B.1.9 Any mechanical testing called for in the contract/order /specification has been carried out in accordance with the Clause 8.3.1.

- B.1.10 All accepted bars have been identified with the Quality Control Manager's stamp and such other marking as will enable the following details to be established: -
 - (1) Manufacturer's Trade Mark
 - (2) Cast number
 - (3) Heat-treatment batch number where heat-treated
 - (4) Test number, where appropriate
 - (5) Specification

APPENDIX 'C'

C. MACRO ETCH TEST FOR CLEANLINESS OF STEEL

C.1 SCOPE

- C.1.1 Macro-etching which is the etching of specimens for examination at low magnification, is a frequently used technique for evaluating steel products such as bars, billets, blooms and forgings.
- C.1.2 This method includes a procedure of rating steel specimens by a graded series of photographs showing the incidence of certain conditions. The method is limited in application to bars, billets, blooms and forgings of carbon and alloy steels.
- C.1.3 This method may involve hazardous materials, operations and equipments and does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitation prior to use.

C.2 SIGNIFICANCE AND USE

- C.2.1 Macro-etching is used in the steel industrial because it is a simple test that will provide information about the heterogeneity of the sample. The method employs the action of an acid or other corrosive agent to develop the characteristics of a suitably prepared specimen. The name implies that the etched surface is examined visually or at only very low magnification. Macro-etching will show:
 - (1) Variation in the structure such as grain size, dendrites and columnar structure
 - (2) Variations in chemical composition such as segregation, coring, and banding and
 - (3) The presence of discontinuities such as laps seams bursts, pipe and flakes.

C.3 SOLUTION

- C.3.1 The most common solution for macroetching iron and steel is a 1 + 1 mixture by volume of concentrated hydrochloric acid (HCl) and water. The acid need not to be reagent grade. Commercial hydrochloric acid, also known as Muriatic Acid, is satisfactory. The solution should be clear and free from scum. It should be used hot 160 to 180°F (71 to 82°C). The solution must be used in fume hood or some other means of carrying off the corrosive fumes must be provided. The solution may be heated without serious change in concentration. The solution may be reused if it has not become excessively contaminated or weakened. A second solution favoured by some as giving a clear structure is composed of concentrated HCl 38 volume %, concentrated sulphuric acid (H₂SO₄)-12 volume % and water (H₂O)- 50 volume %.
- C.3.1.1 Caution Observe caution in mixing. The acids are strong and can cause serious chemical burns, add acid slowly to water mix and etch in fume hard.

C.3.2 AMMONIUM PERSULPHATE SOLUTION

C.3.2.1 A 10 to 20% solution in water is used primarily on longitudinal section to detect certain types of ghost lines, segregations, flow lines etc. A freshly made solution is necessary. Solution should be swabbed on the finished surface at room temperature. Inspection should be done while wet.

C.3.3 NITRIC ACID

C.3.3.1 A 5% nitric acid solution (HNO₃) in methanol or water is used to detect local overstraining, grinding, cracks over-heated areas and depth of carburized or decarburised surface zones. The use of this reagent necessitate a smooth surface. The reagent is used at room temperature. Many other reagents have been used for special application when the use of a reagent other than those described in this para is desired, it should be by agreement between the purchaser and the manufacturers.

C.4 <u>SAMPLING</u>

C.4.1 When using macro-etching as an inspection procedure, sampling should be done in early stage of manufacturing so that if the material proves faulty, the minimum amount of unnecessary work is done. However, the sample should not be taken so early that further working can introduce serious defects. For example the sample is usually taken after ingot breakdown and after most chances of bursts or flakes has

passed. Billets or blooms going into small sizes are sampled after initial break down. Material going into forging billets or die blocks is sampled near finish size. Forgings may be sampled before or after heat treatment. Sampling may be done systematically or in a random basis.

- F.4.1.1 Normally the samples are disks, cut from the ends of bars or billets. Enough material should be discarded before taking the sample to eliminate any extraneous effects of rolling such as fish tails. Samples may be cold cut by any convenient means, saws and abrasive cut off wheels are particularly effective. Torch cutting or other hot cutting while materially affect the structure of the sample may be used only when necessary to remove a larger piece prior to cutting to size by cold methods. Sufficient torch cut surface should be removed to eliminate the effect of torch cutting. The macro etch test as applied to the inspection of steel product of this specification is carried out on sample disks, usually 13 to 25 mm in thickness. Disks or specimen are usually cut to reveal a transverse surface but the requirements of the specimen, contract or order may include the preparation and examination of a longitudinal surface.
- C.4.1.2 When the test is conducted on single specimen the purchaser may specify that the sample disks be cut to represent both ends or one end only of the specimen.
- C.4.1.3 When the test is conducted on a number of specimens made from a heat of steel, the purchaser may require that each specimen be individually tested, or a representative method of sampling may be agreed upon by the manufacturer and the purchaser.
- C.4.1.4 For the indication of certain internal types of discontinuities such as thermal cracks or flakes, the purchaser may specify that disks for macro-etch inspection be taken a certain minimum distance from the ends of the specimen. In the case of forgings depending upon prior agreement either by allowing excess metal for discard on the end or ends of the forging or by forging in multiple lengths and removing the test disks between individual pieces when cutting up the multiple forging.

C.5 PREPARTION

C.5.1 Sample preparation need not be elaborated. Any method of presenting a smooth surface with a minimum amount of cold work will be satisfactory. Disks may be faced on a lathe or a sharper. The usual procedure is to take a roughing cut, then a finish cut. This will generate a smooth surface and remove cold work from prior

operations. Sharp tools are necessary to produce a good specimen. Grinding which also may be used, is usually conducted in the same manner, using free cutting wheels and light finished cuts. When fine detail is required, the specimen should be finished with metallographic papers.

After surface preparation, the surface to be etched should be clean. C.5.1.1Any grease, oil, or other residue will produce uneven attack and must be removed. It may be necessary to use solvents to clean the surface and once cleaned, care should be taken to avoid touching or otherwise contaminating the surface.

Large cross sections may be cut into smaller pieces to facilitate handling and to comply with safety requirements. The sectioning of

the large specimens should be done so as not to disturb the centre

portion of the material.

C.6**PROCEDURE**

C.5.1.2

- Macro-etching is carried out in containers which must be fairly C.6.1 resistant to the attack of the etching agent. Small pieces may be etched in a glass or porcelain vessel of the types commonly available in the laboratory. Large disks are treated in corrosion resistant alloy vessels, various types of ceramic pots, rubber lined kettles or wooden tanks. When metal is used, lead is most common for resisting sulphuric acid solution and high Nickel iron or high silicon iron for Hydrochloric acid. For resisting sulphuric acid or Hydrochloric acid or both, a Nickel Molybdenum alloy can be used. It is important that the prepared faces or the edges of the disks being etched are not in contact with each other or with the vessel, if it is metallic Setting up electrolytic couples by contact of the two metals in an electrolyte causes extremely uneven and misleading attack. The resin in the wood to make tank sometimes act as uncertain and uneven inhibitors resulting in unsatisfactory etched disks.
 - Mix the solution and place in a corrosion resistant tray or disk and C.6.1.1bring to operating temperature before starting any etching. Do not place specimen in a cold solution and then heat to temperature. The specimen may be placed directly into the solution, but the best practice is to place the specimen in corrosion resistant baskets or on corrosion resistant supports such as glass rods, underneath the specimen to raise it off the bottom of the disk. Maintain a sufficient volume of solution in the vessel to cover the specimen with a layer of at least 25 mm of liquid. When etching is complete remove the specimen from the solution, taking great care not to mar the specimen

surface. Remove the smut that forms on the specimen surface by scrubbing the surface with a stiff brush under hot running water. This brush may be natural vegetable or synthetic fibre but not metal. After the smut is remove, rinse the specimen in hot running water and blow dry with compressed air. The specimen should not be blotted dry. Dried surface may be protected with oil or a transparent lacquer. The best time for examination's is however, immediately after drying.

C.6.1.2 The time of etching will vary depending on composition, size, whether pre-heated or not, etc. Etch the specimens to reveal the structure clearly and then remove from the bath. Overeaching can lead to mis-interpretation. Generally speaking 5 to 30 minutes will be sufficient.

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