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Mazdoor Kisan Shakti Sangathan
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"पुराने को छोड नये के तरफ"
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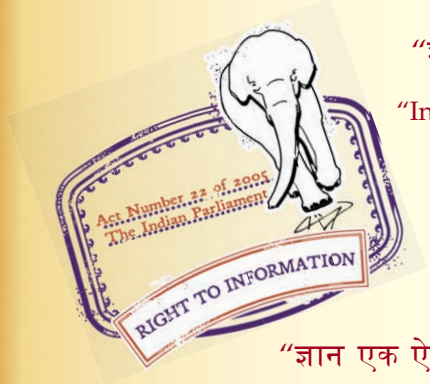
"Step Out From the Old to the New"

IS 13015 (1991): Macroetch testing, inspection and rating of steel products [MTD 22: Metallography and Heat Treatment]



"ज्ञान से एक नये भारत का निर्माण"
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"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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IS 13015 : 1991
(Reaffirmed 2003)

Indian Standard

STEEL PRODUCTS — MACROETCH TESTING, INSPECTION AND RATING — SPECIFICATION

UDC 669·14·015 : 620·183·255

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 1991

Price Group 4

Metallography and Heat Treatment Sectional Committee, MTD 22

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Metallography and Heat Treatment Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard has been prepared with a view to guide the industry for evaluation of steel products by macroetching.

In the preparation of this standard assistance has been derived from ASTM E-381 'Standard methods of macroetch testing, inspection and rating, steel products, comprising bars, billets, blooms and forgings and JIS G 0553 : 1983 'Macrostructure detecting method for steels'.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding of numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

STEEL PRODUCTS — MACROETCH TESTING, INSPECTION AND RATING — SPECIFICATION

1 SCOPE

1.1 This specification deals with the evaluation of steel products such as bars, billets, blooms and forgings by macroetching.

2 GENERAL USES

2.1 Significance

The data obtained from macro examination of steel provides important indications regarding heterogeneity of a material and consequently determine its serviceability. Macroetch test method is used as an evaluating technique for revealing:

a) variations in structure, such as grain size, flow lines, columnar structure, dendrites, etc;

b) variations in chemical composition as evidenced by segregation, coring, etc; and

c) presence of discontinuities and voids produced during manufacture such as seams, laps, porosity, flakes, bursts, rupture cracks, etc.

2.2 Applications

Macroetching is done by etching a suitably prepared specimen by an appropriate reagent. This test is used extensively for quality control in steel industry, and to determine the 'tone' of a heat in billets with respect to inclusions, segregation and structure. Steels react differently to etching reagents because of variations in chemical composition, method of manufacture, heat-treatment and many other variables. Therefore, the establishment of general standards for acceptance or rejection must be considered relative to the part the indication occurs in.

3 SAMPLING

3.1 Sampling Plan

When in accordance with the requirements of the enquiry or contract, forgings, billets, blooms, etc, are subjected to macroetch testing and inspection, the sampling plan shall be made as per the agreement between the manufacturer and the purchaser and shall include the following:

a) the stage of manufacture at which the test shall be carried out;

b) location and number of specimen per test to be examined;

c) the type, size, number, location and orientation of heterogeneities that are considered injurious.

NOTE—When note specified the procedure of the test may be selected by the manufacture to satisfy the relevant requirements.

3.2 Selection of Samples

By macroetching appropriately selected samples/sections at an early stage of manufacture, defective material may be detected early thereby avoiding wasteful end product. Sampling may be done after ingot breakdown and after most chances of bursts and flakes occurring have passed. While stages of examination shall depend on agreement between manufacturers and purchasers, it is usually carried out at semi-finished and/or finished stage. Sampling may be done systematically or on a random basis.

3.3 Billets, Blooms and Hot-Rolled Products

Discs are usually cut from these products near the ends covering entire cross section of the product.

3.4 Forgings

Discs may be cut transverse to the long dimension to reveal defects like cracks, bursts, flakes, etc. For revealing flowlines, forgings should be cut parallel to the long dimension.

NOTE — The discs cut from hot-rolled products or forgings should not be too large to handle, usually 12 to 25 mm thickness and maximum 75 cm^2 in cross section will do.

4 PREPARATION OF SAMPLE

4.0 Sampling preparation need not be elaborate. Any method for preparing a smooth surface with a minimum amount of cold work will be satisfactory.

4.1 For hot mill products such as bars, billets, etc, the disc cut with parting tool is prepared by facing on a lathe or by grinding. But the specimen must be kept sufficiently cool to prevent heating the surface to an excessively high temperature.

4.2 For inspection purpose, finishing on 120 grit grinding wheel or on No. 00 or No. 000 emery papers should be done, when maximum amount of detail careful preparation may be necessary to provide a smooth surface with a minimum

amount of cold work. This is achieved by polishing the specimen with a series of metallographic papers to obtain the desired smooth polished surface.

It should be ensured that the polished surface of the specimen remains free from contamination with dirt, oil grease, etc.

5 MACROETCHANTS FOR STEEL

Selection of macroetchants for steel depend on the purpose of examination and types of alloy grades (*see* Table 1). The reagents should be of good quality but need not be of analytical grade. The solution should be clean, clear, free from suspended particles, scum, etc. Maximum precautions are to be taken while handling, mixing or heating the chemicals.

6 TESTING METHOD

6.1 Acid Etch

6.1.1 Dilute industrial hydrochloric acid by equal quantity of water (add acid slowly to water) to make an etching reagent and use after heating it to 75 to 85°C in a corrosion resistant container, preferably under a fumehood. Sufficient quantity of the etching reagent should be used to prevent any appreciable change in density on the etched surface.

6.1.2 The test piece shall be placed in the container on some non-reactive support with surface to be tested turned upwards or held vertically avoiding contact with between individual test pieces and maintaining, as far as possible reagent temperature constant. Test pieces may be preheated in hot water before immersion in the macroetchant. The standard preheating temperature should be between 75 to 85°C.

6.1.3 The time of etching shall depend on development of desired structure which varies with composition and condition of the metal. (Normalised, hardended, etc) and the temperature of the etching reagent. The progress of etching should be closely watched and etching stopped when desired structural details are revealed. Generally a light etch is better than a heavy. Overetching can often lead to misinterpretation. The standard etching time and temperature is best determined by trial.

6.1.4 When etching is completed, the specimen is removed from the container and washed in warm or running water. During rinsing, all the traces of smut are removed by careful brushing of the etched surface. The specimen is then neutralized in a suitable solution, washed in running water once again and then dried in a blast of hot air from an ordinary hair drier. Rinsing of the etched surface with alcohol prior to the hot air blast shall provide superior and effective drying. The surface is to be examined immediately after drying and observations are to be noted. Macroetched surfaces may be preserved by coating the surface with a thin layer of clear lacquer.

6.1.5 In case of a large specimen, swabbing may be the only practical method of macroetching. A web of cotton held in stainless steel or nickel tongs is saturated with the etchant and swept over the surface of the specimen. After initial wetting, the swab should be kept saturated with the etchant and sweeping over the surface of the specimen be done frequently to renew the solution. When the desired structure is suitably developed, the specimen will be rinsed by pouring water over the specimen and should be swabbed by alcohol. The specimen will be inspected immediately after drying.

6.2 Etching in Ammonium Persulphate Solution

A freshly prepared 10 to 20 percent solution in water is necessary. The solution is swabbed on the finished surface of the specimen at room temperature. Swabbing is to be continued till desired surface structure is developed (i.e. ghost lines, flow lines, segregation, etc; are revealed). Inspection should be done while wet.

Table 1 Section of Macroetchants

Sl No.	Etchant	Composition		Temperature	Alloy Types Applicable
i)	Hydrochloric acid	HCl (conc) H_2O	50 ml 50 ml	70-85°C	General purpose
ii)	Nitric acid	HNO_3 (conc) H_2O	5 ml 95 ml	Room temperature	High alloy steel (for large surfaces)
iii)	Mixed acid	HCl (conc) H_2SO_4 (conc) H_2O	38 ml 12 ml 50 ml	70-85°C	Plain & alloy steel cutlery (12% Cr) steel
iv)	Ammonium persulphate	$NH_4S_2O_8$ H_2O	10-20 g 100 ml	Room temperature	Grain size weldment of plain & low alloy steels
v)	Humfrey's reagent (Copper-ammonium chloride)	$CuNH_4Cl$ HCl H_2O	120 g 50 ml 1000 ml	Room temperature	Produces strong relief effect to reveal segregation

6.3 Etching in Ammonium Copper Chloride Solution

This method is used for detecting segregation and other defects. Particularly for material with comparatively large section, 100 to 350 g of industrial ammonium copper chloride is added to 1 000 ml of water to make an etching reagent. Etching shall be effected at room temperature and a sufficient quantity of the etching reagent shall be used to prevent any appreciable change in density on the etched surface. A fresh solution should be used, as a rule, for each test.

The specimen, carefully prepared to a finely polished etching surfaces by polishing through successive grades of metallographic emery papers, is to be placed in the etching reagent with the surface to be tested turned upwards or held vertically immersed therein in such a manner that a sufficient amount of etching reagent will remain on its surface.

As etching progresses there will be a precipitation of copper on the surface. The copper which is allowed to deposit undisturbed for about 5 minutes shall be removed with a brush or a sheet of cloth. The process is repeated till the desired condition is attained.

After etching, the test piece is washed in running water. All the traces of corrosive products are brushed off from the surface. After neutralizing and washing in warm/running water, the test piece is dried quickly in a jet of hot air to make immediate careful visual examination.

7 EXAMINATION OF SPECIMENS

7.1 After drying (except when using ammonium persulphate etchant), the macrostructure shall be examined carefully. A series of photographs of etched specimens are presented in two groups.

7.1.1 *Plate I*

Grades series for three conditions:

a) Sub-surface conditions,

b) Random conditions, and

c) Centre segregation.

7.1.2 *Plate II*

Consists of a group of photographs showing various other conditions like pattern, flute, crack, gassy, butt tears, splash, flakes.

While examining, the macroetched specimens, the appearance of the specimen is compared to the photographs in Plate I and the photograph number most nearly representing the appearance in each of the series is reported.

The specimen will also be compared against Plate II, and the presence of any of the conditions in Plate II will be reported.

It may be noted that the presence of ingot pattern is acceptable in any degree, but defects like flute cracks, gassy, butt tears, flakes are not acceptable in any degree.

7.1.3 The general standard of acceptance will be relative to end use considerations. The acceptability rating shall be decided between the purchaser and the producer.

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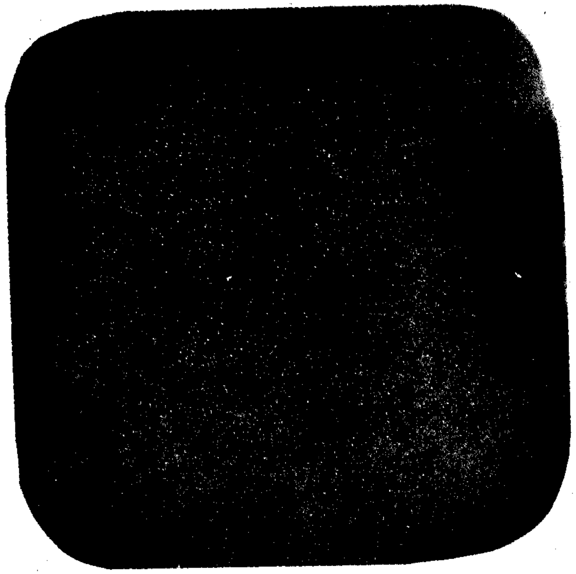

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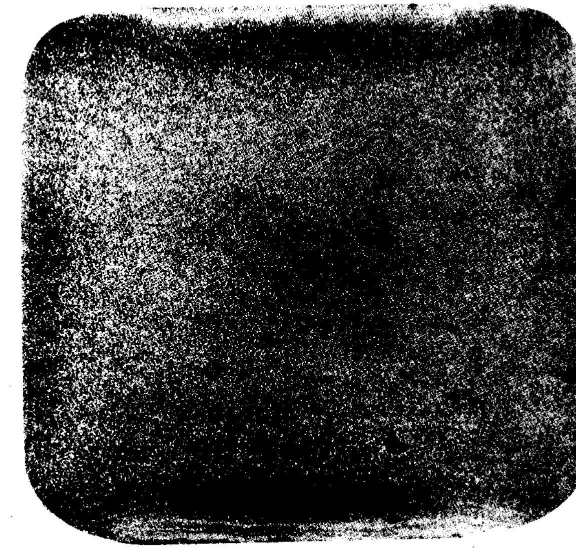
R-2



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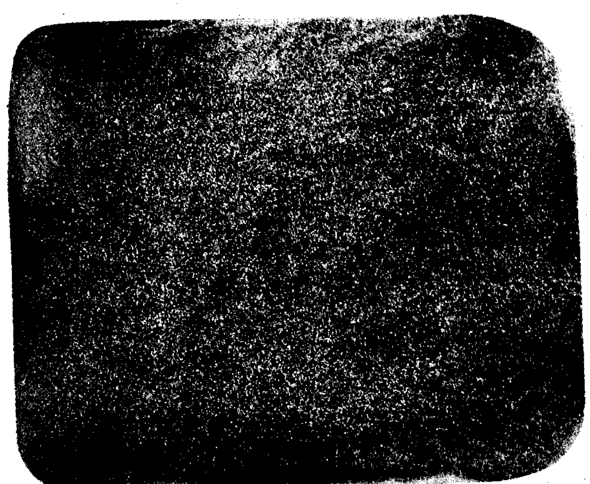

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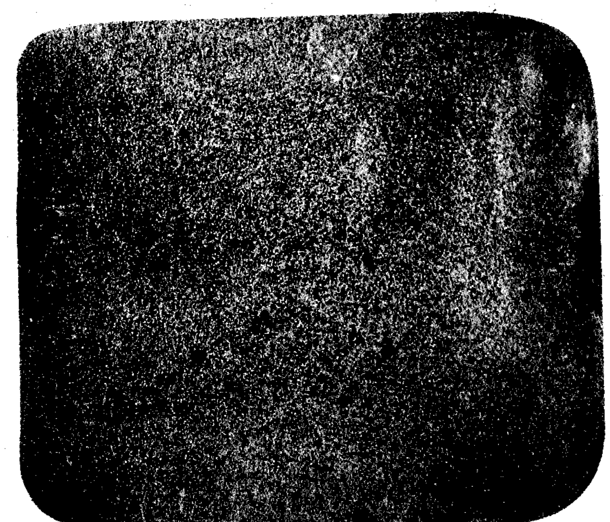
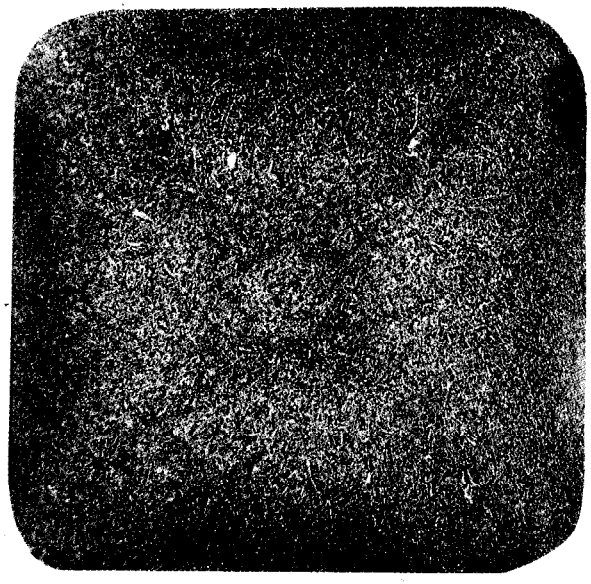


R-5

RANDOM CONDITIONS

PLATE 1 *(continued)*



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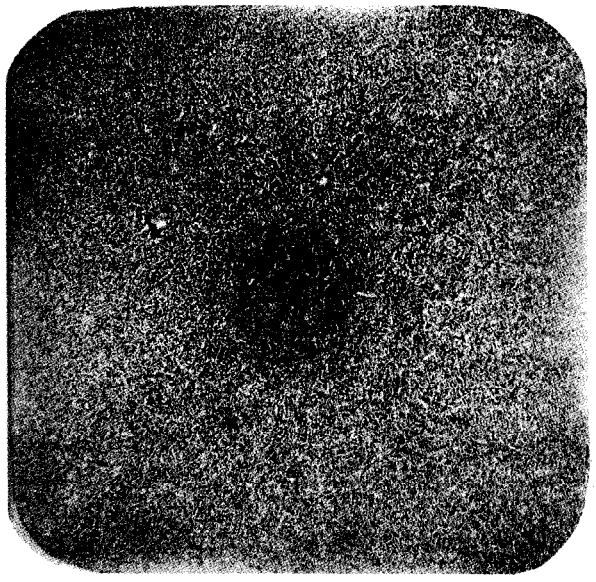

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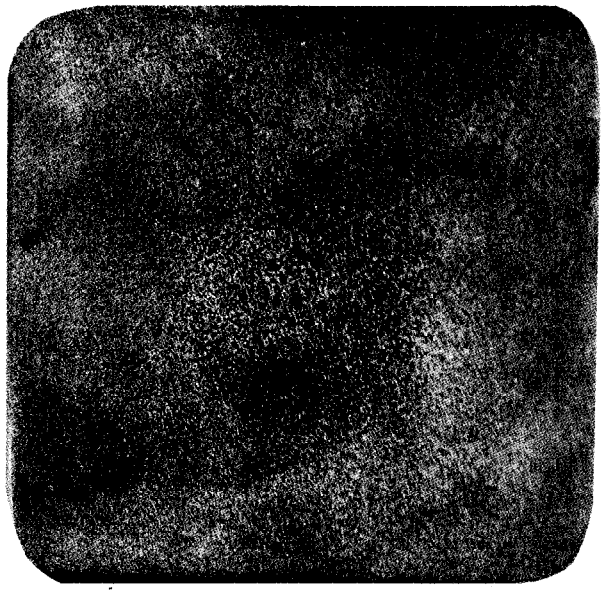

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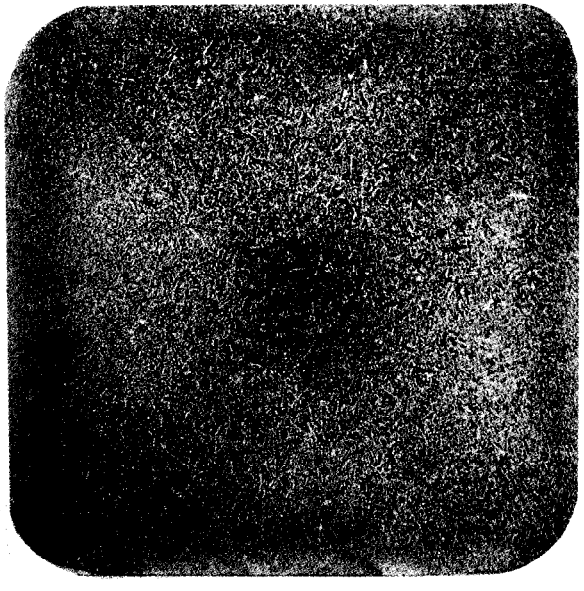

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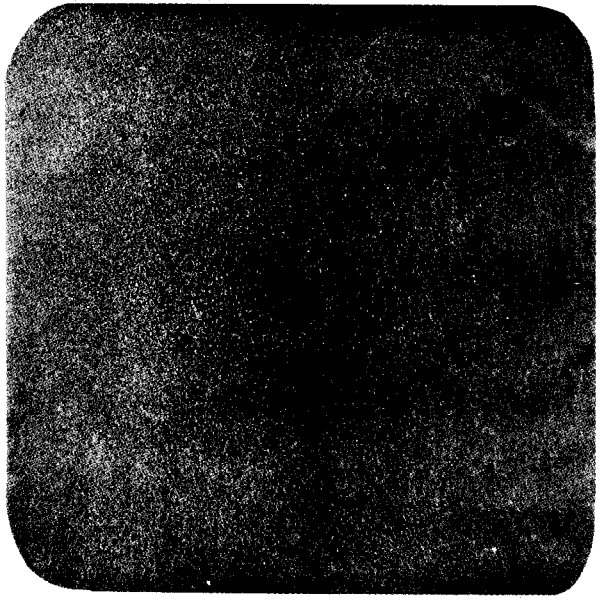
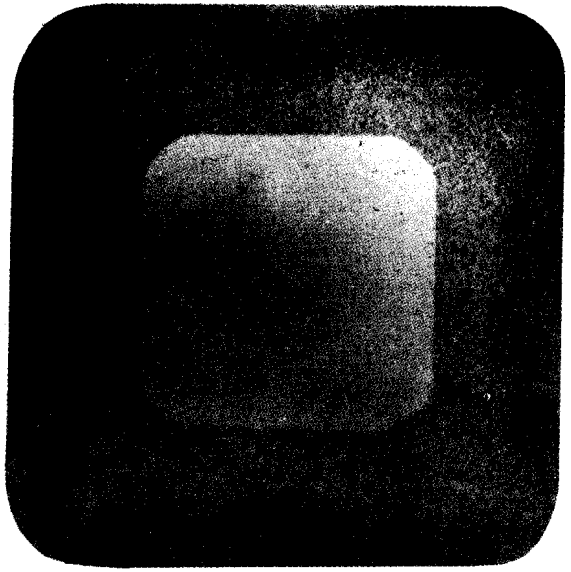


C-5

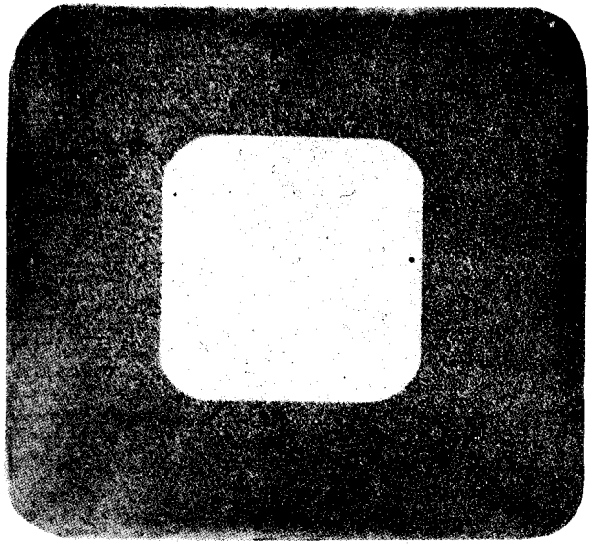
CENTRE SEGREGATION

PLATE I *(continued)*



S-1

S-2



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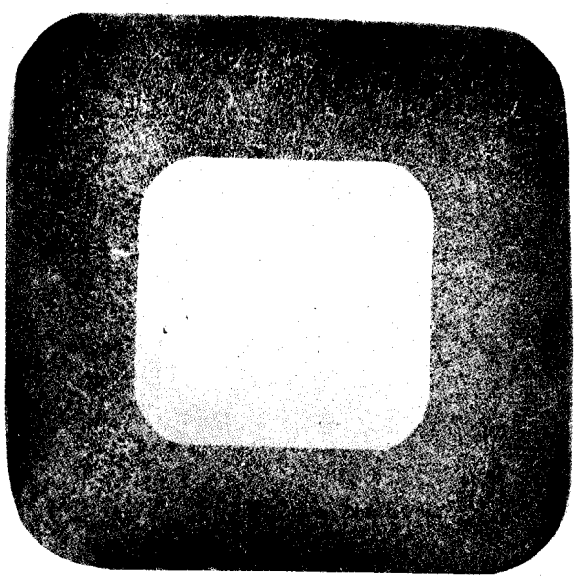

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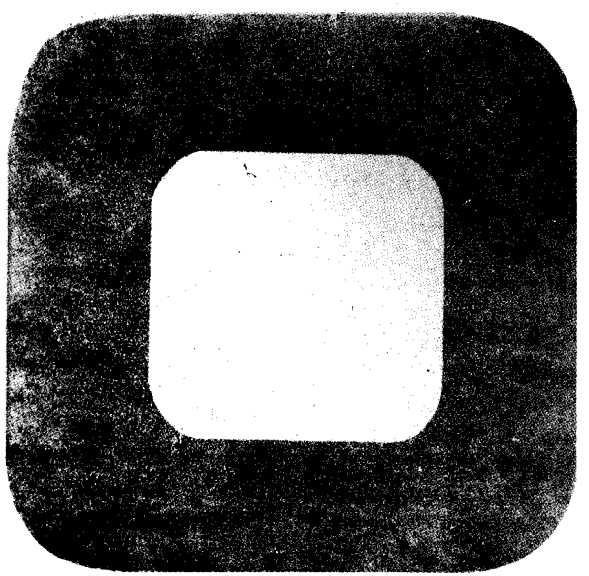
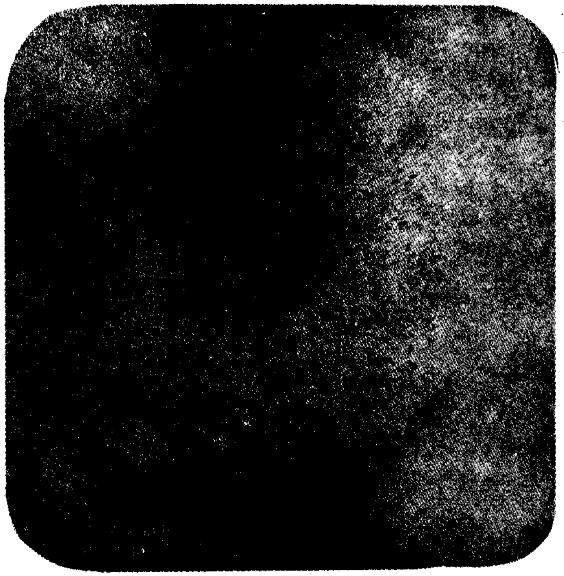


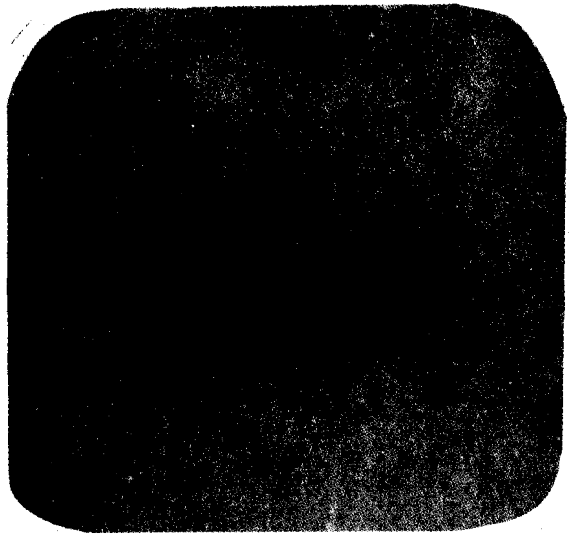
S-5

SUB-SURFACE CONDITIONS

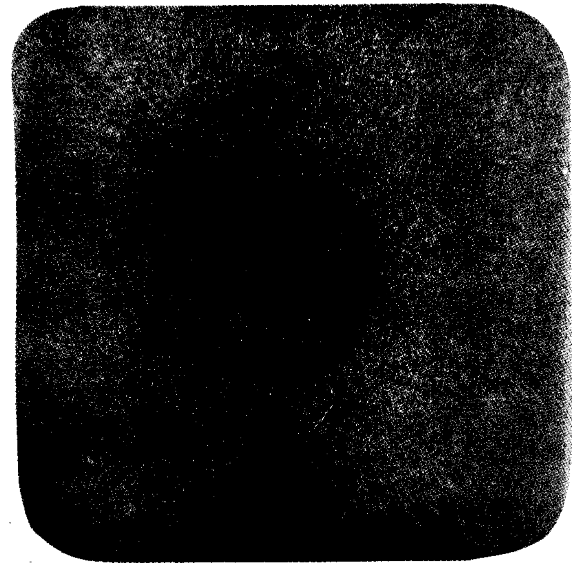
PLATE I *(concluded)*



FLAKES

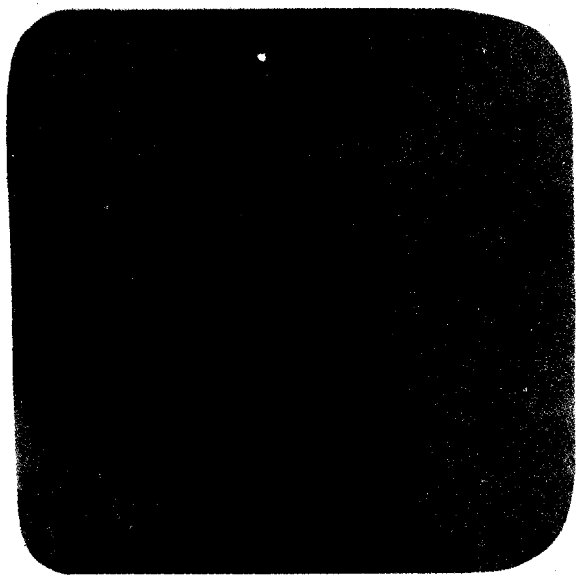


GASSY

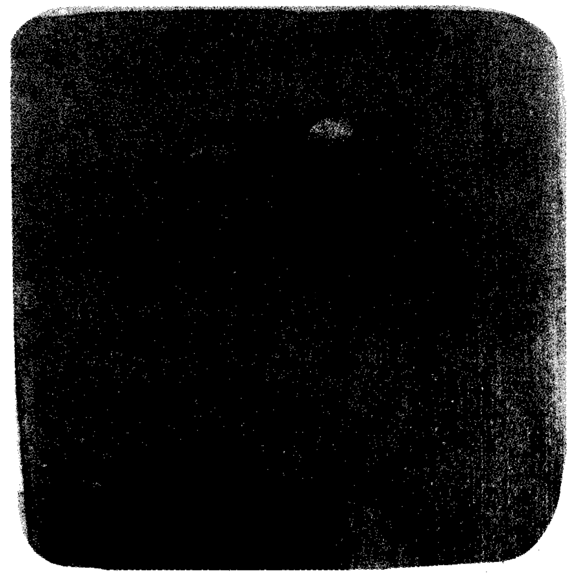


PATTERN

FLUTE CRACKS



BUTT TEARS



SPLASH

PLATE II

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Indian Standards are reviewed periodically and revised, when necessary and amendments, if any, are issued from time to time. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition. Comments on this Indian Standard may be sent in BIS giving the foll owing reference :

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