भारतीय मानक

IS 13484 : 2024

Indian Standard

ताँबा और ताँबा मिश्रण के लिए स्थूल रसोत्कीर्णन — परीक्षण पद्धति

(पहला पुनरीक्षण)

Method for Macroetch Test for Copper and Copper Alloys — Method of Test

(First Revision)

ICS 77.080.20

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

June 2024 **Price Group 4**

Metallography and Heat-Treatment Sectional Committee, MTD 22

FOREWORD

This Indian Standard (First Revision) 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 was first published in 1992. This revision has been brought out to bring the standard in the latest style and format of the Indian Standards.

Macroetching is used to reveal the heterogeneity of metals and alloys. Metallographic specimens and chemical analysis will provide the necessary detailed information about specific localities but they cannot give data about variation from one place to another unless an inordinate large number of specimens are taken.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the result of a test or analysis made in accordance with this standard, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical-values (*second revision*)'.

Indian Standard

METHOD FOR MACROETCH TEST FOR COPPER AND COPPER ALLOYS — METHOD OF TEST

(First Revision)

1 SCOPE

Macroetching is the etching of specimen for examination with unaided eye at low magnifications and is used to reveal the macrostructure of copper and copper alloy products, such as bars, billets, forgings and castings. The present procedure is intended to be followed for copper and copper alloys, showing characteristic structure and incidence of certain processing defects and is applicable to wrought and cast products.

2 SIGNIFICANCE

Macroetching is a simple test which provides information about the heterogeneity of the sample. It will show: (a) variation in structure such as grain size, flowlines columnar structure dendrite, etc; (b) presence of macro segregation and banding, coring and inclusion; and (c) the presence of discontinuities and voids, such as lamps, porosity, extrusion rupture, seams and bursts etc.

3 SAMPLING

3.1 When using macroetching as an inspection procedure, sampling should be done in an early stage of manufacturing so that if the material proves defective, the minimum amount of unnecessary work is done. However, the sample should not be taken so early that further working can introduce serious defects. The sample is usually taken after ingot breakdown and after most chances of flaking and bursting have passed. Billets or blooms going into small sizes are sampled after initial breakdown. Materials going into forging billets or other products are sampled at near finish size, sampling may be done systematically or on a random basis.

3.1.1 Some common methods of sampling are listed as follows.

3.1.1.1 *Billets, bloom and hot-rolled products*

Discs are usually cut from these products near the end, but fish tails should be avoided. Discs from large blooms can be cut into smaller pieces for ease in handling.

3.1.2 *Forgings and Extrusion*

Discs, cut transverse to the long dimension, are used for examining bursts, voids, etc. Forging should be cut parallel to flash lines to show flow lines. Macroetching of an unprepared specimen shows surface defects, such as shuts, flats, seams, etc.

3.1.3 *Sheets and Plates*

While looking for defects in sheets, as large a sample as possible should be taken. When seeking information on lamination, a transverse section is used.

3.1.4 *Castings*

Specimen should be cut from different regions depending upon the features sought.

3.2 When macroetching is used to solve a problem, then the problem itself largely dictates the sample location on the work pieces and the stage of manufacture; for example, when looking for pipe, the sample should represent for top of the ingot, or when looking for burst, the sample should be taken as soon after hot working as possible.

3.3 Sample may be cold cut by any convenient means. Saw and abrasive cut off wheels and EDS cutting are particularly effective. The surface to be examined should be properly prepared before macroetching.

4 SPECIMEN PREPARATION

4.1 Sample preparation need not be elaborate. Any method of presenting a smooth surface with a minimum amount of cold work will be satisfactory. Discs may be faced on a lathe or a shaper. The usual procedure is to take a roughing cut, then a finish cut. This will generate a smooth surface and remove cold work layer from prior operations. Grinding may also be employed using a free cutting wheel and light finishing cut. When fine details are required, the specimen should be finished through the series of metallographic paper.

4.2 After surface preparation, the sample should be cleaned carefully with suitable solvents. Any grease, oil or other residue left on the surface will result in uneven attack during macroetching. Care should be taken not to touch the sample surface or contaminate it in any other way after it has been cleaned.

5 SOLUTIONS

5.1 The most common solutions for macroetching copper and copper alloys are given in Table 1.

Table 1 Macroetchants for Copper and Copper Alloys

(*Clauses* 5.1 *and* 6)

Sl No.	Alloys	Composition		Procedure	Comments
(1)	(2)	(3)		(4)	(5)
i)	Cu and all brasses	HNO_3 H_2O	10 ml 90 ml	Immerse specimen in solution at room temperature for a few minutes. Rinse in water and swab with alcohol and dry	Highlights grains and cracks
ii)	Cu and all brasses	HNO_3 H_2O	50 ml 50 ml	As above	Brings out grain, contrast. Pitting occurs unless the etchant is agitated. Aluminium bronzes may from smut which can be removed by brief immersion in concentrated HNO_3
iii)	Cu and all brasses	HCl $FeCl_3$ H_2O or ethanol	30 ml 10 ml 120 ml	As above	Good grain contrast
iv)	Cu and high Cu alloys, Phosphor bronzes and tin bronzes	$K_2Cr_2O_7$ Saturated solution of NaCl H_2SO_4 H_2O	2 g 2 ml 8 ml 100 ml	Immerse specimen in solution at room temperature for 15 min to 30 min then swab with fresh solution. Rinse in warm water and dry	Highlights grain boundaries and oxide inclusions
v)	All	HNO_3 $AgNO_3$ H_2O	50 ml 0.5 ml 50 ml	Immerse specimen in solution at room temperature. Rinse in warm water and dry	Brilliant deep etch
vi)	Brass	20 percent acetic acid 5 percent chromic acid 10 percent $FeCl_2$ in H_2O	20 ml 10 ml 5 ml	As above	Strain lines
vii)	Silicon brass or bronze	Cr_2O_3 NH_4Cl HNO_3 (conc) H_2SO_4 (conc) H_2O	40 g 7.5 g 50 ml 8 ml 100 ml	Immerse specimen in solution at room temperature, rinse in warm water and dry	General structure

5.2 Caution must be observed in mixing the acids as many of the acids may cause chemical burns. The acid should be added slowly to water. Mixing and etching should be done in a fume hood as many of the solutions are aggressive and may give irritating fumes.

6 PROCEDURE

The specimen should be etched to reveal structure by immersion or by swabbing or by both means. The time of etching will vary depending upon the composition, size and structural conditions. Generally speaking 30 s to 10 min will be sufficient. Etching should be stopped when the preferred structural details have been revealed. Over-etching can lead to misinterpretation and hence light etching is better. Table 1 gives the time for various etchants, but should be used as a guide only. The actual time to develop structure properly may be quite different from that suggested. After etching specimen should be washed in running water and blown dry with compressed air.

7 EXAMINATION OF SPECIMENS

7.1 Any special conditions like segregation, banding, voids, etc, that can be observed should be reported. A low magnification (× 10) stereo microscope can be used for observation if necessary. Normally it is essential to see the surface from various angles to observe flow defects and banding. The macrograin size of forged billets can be compared with specified macrostructures and level of grain size reported.

7.2 The acceptance and rejection criterion for these macrostructures has to be worked out according to the type of alloy and application. Therefore, the basis of macroetching inspection will be a matter of agreement between the manufacturer and the purchaser, if this technique is being used as a quality test.

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Metallography and Heat-Treatment Sectional Committee, MTD 22

Organization	*Representative(s)*
Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad	DR AMIT BHATTACHARJEE **(*Chairperson*)**
Bharat Forge Limited, Pune	SHRI SURESH ARANGI SHRI SAGAR BAPAT (*Alternate*)
Bharat Heavy Electrical Limited, New Delhi	SHRI VEMANA UDAY KUMAR SHRI VARUN PANWAR (*Alternate*)
Defence Metallurgical Research Laboratory, Ministry of Defence, Hyderabad	SHRI CHANDAN MONDAL SHRI VIVEK KUMAR CHANDRAVANSHI (*Alternate*)
Directorate General of Quality Assurance, Ministry of Defence, Ichapur	SHRI P. SUNDHARAJAN DR JANA BHATTACHARAYA ROY (*Alternate*)
Directorate General of Quality Assurance, Ministry of Defence, Tiruchirapalli	SHRI D. C. KAR
Durgapur Steel Plant, Sail Durgapur	SHRI R. S. TIWARI
Hindalco Industries Limited, Mumbai	SHRI PANKAJ WANJARI SHRI MANU SAXENA (*Alternate*)
Hindustan Aeronautics Limited, Bengaluru	SHRI S. SIVARAMKRISHNAN SHRI D. K. DE (*Alternate*)
Indian Institute of Technology Bombay, Mumbai	PROF NITYANANDA PRABHU PROF K. NARASIMHAN (*Alternate*)
Indian Institute of Technology Kanpur, Kanpur	DR ANISH UPADHYAY DR KRISHAN BISWAS (*Alternate*)
Indian Institute of Technology Roorkee, Roorkee	SHRI SAI RAMUDU MEKA SHRI VARUN BAHETI (*Alternate*)
Institute of Indian Foundrymen, New Delhi	SHRI A. K. ANAND
JSW Steel Limited, Raigad	SHRI GOUTAM MUKHERJEE SHRI ARVIND KUMAR DHAKER (*Alternate* I) SHRI KRISHNA RAMAVATH (*Alternate* II)
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National Test House, Kolkata	SHRIMATI SALY C. M. DR P. KANJILAL (*Alternate*)

Organization	*Representative(s)*
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Tata Motors Limited, Pune	SHRI HEMANT MORE SHRI ANOOP TOBY (*Alternate*)
Tata Steel Limited, Jamshedpur	DR MANASHI ADHIKARI DR GOUTAM MUKHOPADHYAY (*Alternate*)
BIS Directorate General	SHRI SANJIV MAINI, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (METALLURGICAL ENGINEERING) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary
SHRIMATI CHALLAKONDA VIDISHA
SCIENTIST 'C'/DEPUTY DIRECTOR
(METALLURGICAL ENGINEERING), BIS

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

This Indian Standard has been developed from Doc No.: MTD 22 (21985).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.gov.in

Regional Offices:		*Telephones*
Central	: 601/A, Konnectus Tower -1, 6th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	2323 7617
Eastern	: 8th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	2367 0012 2320 9474
Northern	: Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	265 9930
Southern	: C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	2254 1442 2254 1216
Western	: Manakalya, 4th Floor, NTH Complex (W Sector), F-10, MIDC, Andheri (East), Mumbai 400093	283 25838

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Published by BIS, New Delhi