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भारतीय मानक ब्यूरो

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भारतीय मानक मसौदा

धातु विज्ञान के नमूने की तैयारी के लिए अभ्यास संहिता
भाग 1 सामान्य सुविधाएँ
(*आईएस 7739(भाग - 1) का पहला पुनरीक्षण*)

Draft Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS
PART 1 GENERAL FEATURES
(*First Revision of IS 7739(Part 1)*)

ICS 77.080.20

Metallography and Heat-Treatment Sectional Committee, MTD 22

Last date for receipt of comments is 08/09/2023

FOREWORD

(*Formal clauses will be added later*)

The primary objective of metallographic examination is to reveal the constituents and the structure of metals and their alloys by means of a metallurgical microscope (light optical or scanning electron). Because of diversity in available equipment, the wide variety of problems encountered, and the personal element, this standard gives for the guidance of the metallographer only those practices which experience has shown are generally satisfactory.

This standard was originally published in 1975. This revision has been brought out to bring the standard in accordance with the latest practices followed in the industry. Also, clause 3, clause 5 and clause 6 has been modified along with addition of clause 3.3 on Research and clause 6.1.4 on Plastic Mountings in this revision.

This part covers only those phases of the preparation of metallographic specimen which vary little with the material while the special features particularly applicable to one metal and its alloys are covered separately in different parts. The other parts of this standard are as follows:

Part II Electrolytic polishing

Part III Etchants for non ferrous materials

Part V Etchants for ferrous materials

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 : 2022 ‘Rules for rounding off numerical values (*second revision*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART 1 GENERAL FEATURES

(*First Revision*)

1 SCOPE

This standard (Part 1) covers the recommended methods of selection, size, cutting, cleaning and mounting of metallographic specimens for microscopic examination.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreement based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 715 : 2002	Coated abrasives — Specification (*fourth revision*)

3 SELECTION OF SPECIMENS

Microstructure has a strong influence on the end properties and successful application of metals and alloys and therefore metallographic examination is important. Proper selection of metallographic specimen (location, orientation) is required for correct interpretation. Number of specimens selected for the study depends on the intent or the purpose of analysis. Metallographic examination may be divided into three classifications:

3.1 General Study or Routine Work

For general study, specimens from locations which are most likely to reveal maximum variation within the material under study should be chosen. In general, for castings, a section cut perpendicular to the surface is prepared as it reveals the variations in the structure from outside to the interior of the casting. Specific zones of the casting may also be chosen in order to compare zones of maximum and minimum segregation effects in the casting. For hot or cold worked metals, both transverse and longitudinal sections should be studied. In some cases, specimens with surfaces parallel to the surface of original product may be prepared. In the examination of strip or wire, specimens should be taken from each end of the coils. Longitudinal section through the center of the specimen in conjunction with transverse section may be prepared.

3.2 Study of Failures

If the cause for a failure is to be studied, the test specimens are generally taken as close as possible to the fracture or to the area where failure has initiated. Specimens should also be taken from a sound area for comparison of structures and properties. Before sectioning the sample, fracture surface should be carefully studied and documented.

3.3 Research Studies

The type of research will determine the extent of specimen selection with respect to location and orientation. Number of chosen samples will be usually higher in case of research studies.

3.4 Cross sections or transverse sections taken perpendicular to the main axis of the material generally reveal the following:

a) Variations is structure from centre to surface,

b) Distribution of non-metallic impurities throughout the section,

c) Decarburization at the surface of a ferrous material,

d) Depth of surface imperfections,

e) Depth of corrosion,

f) Thickness of protective coating, and

g) Structure of protective coating.

3.5 Longitudinal sections, taken parallel to the main axis of the material, generally reveal the following:

a) Extent of deformation in the non-metallic impurities and the inclusion content of steel;

b) Degree of plastic deformation, as shown by grain distortion;

c) Presence or absence of banding in the structure; and

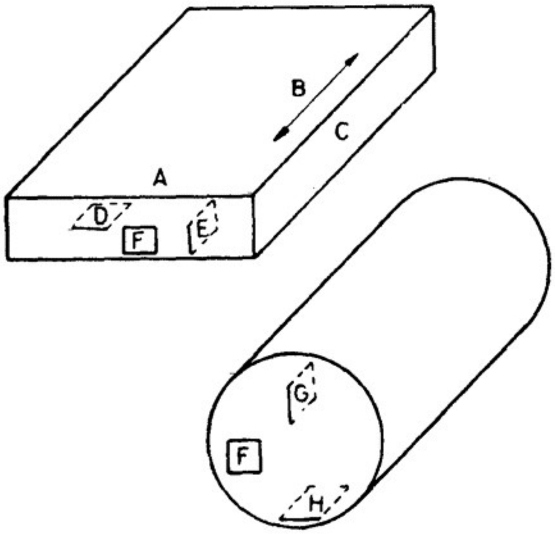
d) In general, microstructure attained with any heat treatment.

3.6 The locations of surfaces examined should always be given in test reports and in any description of any illustrative micrographs, so that it will be entirely clear exactly what sections of the material were used for the examination. A suitable method of indicating surface locations is shown in Fig. 1.

4 SIZE OF SPECIMENS

4.1 The specimen to be polished for metallographic examination should generally be not more than 10 to 25 mm square, or approximately 10 to 25 mm in diameter if the material is round. The height of the specimen should be such that it provides for convenient handing during polishing.

Specimens that are fragile, oddly shaped or too small to be handled readily during polishing, should be mounted for proper microscopic analysis.



Symbol in gram	*Suggested Designation*	*Symbol in Diagram*	*Suggested Designation*
A	Rolled surface	*F*	Transverse section
B	Direction of rolling	*G*	Radial longitudinal section
C	Rolled edge		
D	Longitudinal (or lengthwise) section parallel to rolled surface	*H*	Tangential longitudinal section
E	Longitudinal section perpendicular to rolled surface		

FIG. 1 METHOD OF DESIGNATING LOCATION OF AREA SHOWN IN PHOTOMICROGRAPH

4.2 In the case of wire, strip and other small articles, where it may not be possible to secure specimens, having the dimensions given in **4.1**, the specimens may be suitably mounted for convenient handling (*see* **6**).

5 CUTTING OF SPECIMENS

5.1 The specimen may be cut by a hacksaw, bandsaw or a cutting off wheel. In the case of hard and brittle materials the specimen may be notched and fractured. Flame cutting may be used in order to obtain suitable specimens from large sections. The choice of cut-off blade, lubricant, cooling conditions, and the grade and hardness of metal being cut influences the quality of the cut.

Generally, soft materials are cut with hard bond blade and vice versa. Aluminum oxide abrasive blades are preferred for ferrous metals and silicon carbide blades are preferred for non-ferrous alloys. Abrasive cut-off blades are used for sectioning metals with hardness above about 350 HV. Extremely hard metallic materials may be more effectively cut using diamond impregnated cutting blades. A shear is a type of cutting tool with which a material in the form of wire, sheet, plate or rod is cut between two opposing blades.

5.2 In cutting the metallographic specimen from the main body of the material, care shall be exercised to avoid affecting the structure of the metal. The types of damage to the structure during cutting and the methods employed for removal of the damaged surfaces are:

a) Dragging of the metal by the cutting edge. Lubrication during cutting tends to keep dragging at a minimum.
b) Flame cutting completely alters the structure of the metal at the flame cut edge. If flame cutting is necessary to remove the specimen, it should be cut sufficiently large so that it may be recut to the proper size by some other method.
c) Heating of hardened structure during cutting, thus tempering the material. This heating may be minimized by keeping the work cooled by water during cutting.
d) Where grinding or spark-machining techniques have been used to remove specimens from hard materials, care shall be taken to fully remove the affected layers by prolonged grinding on the coarse paper.

6 MOUNTING OF SPECIMENS

6.1 In some of the instances, like when specimens are too small to be handled readily during polishing or fragile or oddly shaped, should be mounted to ensure a surface satisfactory for microscopical study. The various methods that may be used are discussed in **6.1.1 to 6.1.4.**

6.1.1 *Mechanical Mountings*

Strip and sheet samples are frequently mounted by binding several specimens into a pack held together by two end pieces and two bolts. These end pieces should preferably be of the same hardness as the specimens being mounted, since rounding off the edges of the polished section is often due to the difference in hardness of the specimens and the end holders. The specimens should be tightly bound together to prevent excessive absorption and subsequent exudation of polishing liquids and etchants. Pores may be filled by immersing the pack in molten paraffin; separating the sheets with dense blotting paper may also eliminate this exudation (the blotting paper may become impregnated with abrasive, however, and make polishing difficult). Exudation shall be particularly guarded against if the etchant may attack the lens system of the microscope. The sheets to be examined are frequently alternated with ‘filler’ sheets of a softer alloy in order to secure a more compact specimen and to preserve surface irregularities, but the ‘filler, material shall not react electrolytically with the specimen during etching. Steel sheets mounted with alternate ‘filler’ strips of copper may be etched in the holder since the usual etchant for the steel will not attack the copper. Longitudinal sections of round wire may be mounted by the same method of holders except that only one wire may be mounted in one holder. Alternatively, specimens may also be coated with a layer of epoxy resin before being placed in the clamp in order to minimize the absorption of polishing materials or etchants. Avoid excessive clamping pressure which may otherwise damage the soft specimens.

6.1.2 *Electroplated Mountings*

Small wires, odd shapes, fractures, or specimens of which the edges are to be examined are frequently electroplated before mounting to avoid tearing or rounding off during subsequent polishing. These electroplated specimens are invariably mounted by some other process after plating. Ferrous metals are usually copper plated for this purpose. A flash coat in a copper cyanide bath (Note 1) is first applied, followed by plating in a copper sulphate bath (Note 2). Some laboratories frequently apply the entire coat using the cyanide bath. The precious metals are frequently plated with nickel, gold, or silver, while fractures of many non-ferrous metals are conveniently plated with silver. The plating metals should be somewhat softer than the specimen to avoid differential polishing of the material, that may mask the edge, and should not react electrolytically with the base metal of the specimen. Electroless plating is preferred to electrolytic plating for specimens with rough, porous, or irregular surfaces, as it provides better surface coverage and penetration. Electroless plating solutions can be purchased commercially.

Plating thickness is recommended to be at least 5 microns.

NOTE 1 — For the copper cyanide bath use 22.5 g of cuprous cyanide, 34 g of sodium cyanide and 15 g of sodium carbonate in a litre of water; current density of 0.2 A/dm^2.

NOTE 2 — For the copper sulphate bath use 250 g of copper sulphate crystals and 75 g of concentrated sulphuric acid (sp gr 1.84) in a litre of water; current density of 2 to 4 A/dm^2.

6.1.3 *Cast and Cement-like Mountings*

Some types of specimens are frequently placed in a mounting ring and a mounting medium is cast around them. In selecting the mounting medium, some consideration shall be given to its resistance to attack by the etchants to be used and the effect of the mounting on the specimen in regard to electrolytic attack at the point of contact. Likewise, a thermal effect due to the temperature of the melting point of the mounting shall be given consideration, so that no change occurs in the structure of the specimen to be examined. In general, this classification of mounting may be divided into two fields; fusible metal alloys which are cast to shape, and cement-like mixture which harden to shape.

Castable plastic mounts are usually prepared at room temperature. Some may require external heat source or applied pressure in order to cure. These resins consist of two or more components which must be mixed just prior to use. There are four kinds of castable plastics in common use like acrylic, polyester-acrylic (quartz filled), polyester and epoxy. The cure time of these resins vary from 8-15 min for former two, to 30-60 min to ½-20 hour respectively. The properties of these resins also vary with respect to shrinkage, peak curing temperature, abrasion resistance and transparency. Molds for castable plastics are often simple cups which hold the resin till cure. Handling castable resin requires care to avoid skin problems like dermatitis.

6.1.3.1 Ferrous metals and alloys are occasionally mounted in solder of 50 percent lead and 50 percent tin. An alloy of 52.5 percent bismuth, 31.5 percent lead, and 16 percent tin (melting point 95 to 98°C) is used in some laboratories. Cadmium-zinc eutectic (82.5 percent cadmium, 17.5 percent zinc, melting point 264.5°C) has been found a useful mounting material for zinc castings, since it possesses the same polishing and etching characteristics as zinc. The temperature of the alloy should be kept below 300°C since cadmium oxide fumes are very poisonous.

Sulphur is used as a mounting medium as it has a low melting point (119°C) and resists attack by most cold-etching reagents. It may sometimes cause pitting in steel samples and occasionally tends to tarnish copper alloys.

6.1.3.2 Similar mounting may be done by using plaster of paris, sealing wax, iron-ammonium chloride cements, litharge-glycerin cement, and some dental cements, especially for non-ferrous materials, such as gold, platinum, other precious metals, and low melting-point alloys.

6.1.4 *Thermosetting and Thermoplastic Materials*

Modern plastic of either the thermosetting or thermoplastic types form a class of metallographic mounting materials more or less related to the cast or cement-like materials. In general, they are employed for the same reasons and in much the same manner. It should be noted, however, that specimens of hardened alloys may be softened by mounting in this way, since heating temperatures between 140 and 160°C is required.

6.1.4.1 The thermosetting materials undergo a chemical reaction on application of heat and pressure during the moulding process which-causes them to set up into a more or less permanent state that cannot further be changed by temperatures up to those which cause the material to char. The thermoplastic resins, on the other hand, will flow each time that the proper combination of heat and pressure is applied. This difference between the materials is important and should be recognized. There is little, if any, choice between the materials in so far as their ability to be moulded adequately is concerned. Heating temperatures are around 140 and 180°C and force applied is around 27-30 MPa.

6.1.4.2 The thermosetting resins are usually of the formaldehyde type, whereas the thermoplastic resins comprise the polystyrenes, methacrylates, cellulose-base materials, etc.

6.1.4.3 Because of the fundamental differences in the two classes of materials, the thermoplastic resins are cooled to approximately 75 to 80°C before removal from the mould whereas the thermosetting resins may be removed from the mould as soon as the resin has ‘set up’.

Regardless of the resin used, best results are obtained when 1) the specimen is clean and dry, 2) the cured mount is cooled under full pressure to below 40°C before ejecting from the press ensuring minimal shrinkage gap formation.

6.1.4.4 Comparing these materials from the standpoint of chemical resistance, it is found that the thermosetting resins are decomposed by strong oxidizing acids and strong alkalis but are resistant to other materials, while the thermoplastic resins may somewhat be attacked by strong acids and alkalis and are usually very soluble in organic solvents but are otherwise resistant.

6.1.4.5 The nominal Brinell hardness values of the moulding materials, using a 10-mm ball and 500 kg load, are as follows:

Moulding Material	*Nominal Brinell Hardness*
Thermoplastic	
Polystyrene	19-22
Methyl methacrylate	
Soft	17-20
Hard	27
Thermosetting	
Aniline formaldehyde	30
Phenol-formaldehyde (Bakelite)	42

6.1.4.6 With the exception of the aniline formaldehyde, which is more resistant to abrasion than its hardness would indicate, the remaining materials given in **6.1.4.5** withstand polishing in about the same order as of their hardnesses.

6.1.4.7 Keeping in mind the nature of the thermoplastic resins, it is evident that rushing the polishing operation to the point where the sample gets hot may cause considerable flow in the mounting, a circumstance not encountered with the thermosetting types. This may account for the more rapid polishing away and rounding off encountered in the thermoplastic materials. The edges of steel specimens are not very well supported by mounts of this nature, and the use of a harder material or a steel clamp is preferable when sections are to be examined at the extreme edge.

6.1.4.8 The use of transparent mounting materials, which are thermoplastics, seems justifiable only in those instances where sectioning and polishing to an exact position are necessary and the progress of the work has to be observed.

6.1.4.9 The equipment necessary for forming plastic mountings may be made up from a hydraulic auto jack mounted between foot and header plates with a third plate on the head of the jack to serve as the lower platen.

6.1.4.10 The heating may be accomplished by heated platens or by surrounding the mould with a small electric furnace. When thermoplastics are used, cooling methods shall also be utilized. The best combination is obtained by using steam heat and, where necessary, water cooling.

6.1.4.11 Several precautions should be observed if good results are to be obtained, for it is essential that all the material be heated uniformly to the proper temperature, this being accurately checked by thermometers or pyrometers properly located, and that the proper pressures be utilized.

6.1.4.12 For mounting porous or intricate specimens, vacuum impregnation technique may be used. It is accomplished by placing a completely dry specimen in a mold in a vacuum chamber and then introducing the resin into the mould (through a funnel or stopcock fitted) after the chamber has been evacuated or by having a basin of the resin inside the chamber. A low-viscosity resin can produce good results. Pressure inside the chamber must be above critical vapor pressure of the hardener to avoid boiling away the hardener. After the pressure gets equilibrated, the resin in introduced in the mould and the vacuum is released and air admitted to the chamber. Atmospheric pressure will force the resin into fine pores, cracks, and holes.

7 CLEANING, GRINDING AND POLISHING OF SPECIMENS

7.1 Cleanliness during polishing is essential. All grease and oils should be removed by some suitable organic solvent. Any coating metal which will interfere with the subsequent etching of the base metal should be removed before polishing. Rusted fractures may be cleaned with a diluted solution of ammonium citrate or phosphoric acid. In studying the underlying steel in a galvanized specimen, the zinc coating shall be removed before polishing. The zinc may be removed by solution in cold nitric acid (HNO_3) or in diluted sulphuric acid (H_2SO_4). The HNO_3 method requires care to prevent overheating, since large samples will generate considerable heat. By placing the cleaning container in cold water during the stripping of the zinc, the underlying steel will not be attacked after the removal of zinc. Sometimes appropriate inhibitors can be used along with the acid for stripping of zinc to avoid substrate metal loss.

7.2 The surface of the specimen is first made plane by means of a file or by rough grinding on an abrasive wheel or an emery belt. The specimen should be kept cool by frequently dipping it in water during grinding operations in order to avoid any changes in the structure of the metal at the surface. In non-ferrous metals further precautions may be necessary.

7.2.1 The specimens are further ground (wet or dry) on emery/ silicon carbide papers or abrasive wheels/ discs with successively finer grinding media, until a suitable surface is obtained for polishing. Usually, final grinding is done on abrasive materials with 400 to 600 grits. (For details of grit numbers, *see* IS 715). A comparative chart of grit numbers and trade designations is given in Appendix A.

After all grinding is done, the specimen must be cleaned thoroughly. Ultrasonic cleaning in a water/ soap solution containing a corrosion inhibitor may prove beneficial.

7.2.2 Polishing is usually distinguished from grinding by the use of loose abrasive (<=6 microns) embedded in an appropriately lubricated supporting surface. Choice of abrasive, lubricant, polishing surface support depends on the sample type and investigation objective. Polishing can be divided into rough and fine (final) stages. Rough polishing is often sufficient for routine evaluations like micro-indentation hardness and grain size. Fine polishing is performed with diamond or an oxide slurry step or both. Choice of abrasive depends on the specimen hardness. For example, 1 micron diamond final polish is often sufficient for many grades of steel, however, softer steels and non-ferrous materials often require additional polishing step with an oxide slurry or suspension of SiO_2 or Al_2O_3. Final polishing cloths are generally softer and higher in nap than rough polishing cloths. Therefore, polishing time and force must be kept to a minimum to avoid artifacts such as edge rounding and relief. Careful cleaning is of specimen between stages is mandatory. Ultrasonic cleaning may be effective.

The usual practice is to rotate the specimen by 90° when changing from one grade of abrasive to another. The specimen is gradually rotated while polishing on abrasive wheels/ discs. A thorough washing of the specimen while changing, from one grade of abrasive to the next one is recommended. It is also desirable to ensure that scratches from the previous abrasive are eliminated while polishing on the next one- the exception being the first coarsest abrasive in which case care should be taken to fully remove the scratches from the cutting operation. Optimum pressure should be applied while grinding and polishing to avoid deep scratch marks. For semi-auto and auto polishing, speed of grinding and polishing machines may be optimized for best results depending on the type of material.

Polishing operations maybe either manual or automated methods.

Manual polishing consists of holding the specimen by hand against an abrasive -charged rotating wheel and moving the specimen in a circular path around the wheel against the direction of rotation of the wheel. Specimen should be held firmly in contact with the wheel.

Automated methods can perform both grinding and polishing steps and can accommodate multiple specimens and can result in consistent quality with substantial decrease in preparation time. Here the specimens follow an epicycloid path while rotating over the wheel. In some, it can rotate on its own axis as well. Resulting scratch pattern consists of randomly oriented arcs and deciding when the previous scratches have been removed is difficult to ascertain than in unidirectional manual grinding. Applied load in automated machines may be adjusted through the available user interface.

In case of coated materials, moreover for thin coatings, technique of taper mount can be used with the plane of polish at a small angle to the plane of surface

NOTE — Specimens sometimes have adherent debris, oxidation or corrosion product. Either the underlying surface is important or the adherent matter. In either case cleaning of the surface or collection of adherent matter is important. For adherent matter analysis, the surface can be shot blasted, wire brushed, ground, scraped off, or may even use cellulose acetate replica tape (moisten with acetone and press against the debris-coated surface, dry and strip off the tape). When underlying surface is important for analysis, one may use compressed air blast to remove loosely adherent material, soft camel -hair brush. Else one can try aqueous solutions, organic solvents, or alcohol with an ultrasonic cleaner or repeated replication technique. Electrolytic cleaning is yet another approach wherein an inert material is used as an anode while the specimen is made the cathode in the electrolytic cell. Adequate safety precaution must be taken like using fume hood etc for such cleaning practices. A number of proprietary rust removal solutions are also available which can be used suitably. Argon-ion bombardment or glow discharge method requiring special equipment can also be applied.

NOTE — Post cleaning, polishing or etching, specimens may need to be preserved either for short term or long term. A vacuum desiccator may be used for short term preservation. For long term, clear lacquer coat maybe applied, and specimen may be placed in a polymeric container or wrapped up in tissue or cotton and placed in protective box. Appropriate solvent can be used for removing lacquer is required.

6.2.3 The specimens may also be prepared by electropolishing. For details about this process reference may be made to Part 2 of this code.

APPENDIX A

(*Clause* 7.2.1)

COMPARATIVE CHART OF GRIT NUMBERS (APPROXIMATE) OF ABRASIVE GRAINS

Aluminium Oxide Silicon and Garnet		*Flint*		*Class*		*Corundum*		*Emery*		*Trade Designation*
IS Grit Number	BS grade number	IS Grit Number	BS grade number	IS Grit Number	BS grade number	IS Grit Number	BS grade number	IS Grit Number	BS grade number	
14	14	-	-	-	-	-	-	-	-	-
16	16	-	-	-	-	-	-	-	-	-
24	24	24	3	24	3	24	-	24	-	Extra Coarse
30	30	30	$2\frac{1}{2}$	30	$2\frac{1}{2}$	30	-	30	-	Extra Coarse
36	36	36	2	36	-	36	-	36	3	Coarse
40	46	40	$1\frac{1}{2}$	40	S2	40	-	40	$2\frac{1}{2}$	Coarse
50	54	50	1	50	M2	50	-	50	2	Medium Coarse
60	60	60	$\frac{1}{2}$	60	-	60	-	60	$1\frac{1}{2}$	Medium
80	80	80	-	80	-	80	-	80	1	Medium
100	100	100	0	100	F2	100	-	100	F	Medium Fine
120	120	120	00	120	$1\frac{1}{2}$	120	-	120	FF	Fine
150	150	150	-	150	1	150	-	150	-	Fine
180	180	180	-	180	0	180	-	180	0	Extra Fine
220	200	-		-	-	-	-	-	-	-

NOTE — Grits 240 and finer come under the sub-sieve range and as limits for these cannot be set on common silk test ' sieves, the grain sizes shall conform to general commercial grading, and it is recommended that the sedimentation process be adopted for their analysis.