
**Corrosion of metals and alloys —
Exfoliation corrosion testing of aluminium
alloys**

*Corrosion des métaux et alliages — Essai de corrosion feuilletante des
alliages d'aluminium*



Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11881 was prepared by ISO Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

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Corrosion of metals and alloys — Exfoliation corrosion testing of aluminium alloys

1 Scope

WARNING: This International Standard may involve hazardous materials, operations and equipment. It does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1.1 This International Standard describes procedures for constant immersion accelerated exfoliation corrosion testing of aluminium alloys primarily for the purpose of research, development and quality control.

1.2 It covers aspects of the corrosive solution, specimen preparation, exposure, inspection and interpretation of test results.

1.3 This International Standard is applicable primarily to the testing of wrought aluminium alloys in the form of semi-finished mill products and parts produced both from conventional ingot metallurgy processes and from powder metallurgy processes, as well as aluminium alloy metal matrix composites including those produced by mechanical alloying.

1.4 It can also be used for testing ingots and cast aluminium alloys when oriented structures such as columnar grains or striated segregations are present.

1.5 The results of these tests are most applicable to research studies of trends in alloy development and should not be considered as an absolute criterion of the resistance to exfoliation. When these tests are used for production control of exfoliation-resistant materials, limits of acceptable performance should be the subject of an agreement between concerned parties.

2 Normative references

The following standards contain provisions which, through reference in the text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 8044:—¹⁾, *Corrosion of metals and alloys — Basic terms and definitions*.

ISO 11846:1995, *Corrosion of metals and alloys — Determination of resistance to intergranular corrosion of solution heat-treatable aluminium alloys*.

¹⁾ To be published. (Revision of ISO 8044:1989)

3 Definitions

For the purposes of this International Standard the following definition as well as those given in ISO 8044 applies.

3.1 exfoliation corrosion

stratified form of subsurface corrosion of susceptible wrought alloy mill products having a highly directional grain structure, accompanied by detachment of separate layers from the body of the material, formation of cracks and finally possible complete layer-by-layer disintegration of the metal (see also the definition of "layer corrosion" in ISO 8044)

NOTE Exfoliation generally proceeds along grain boundaries, but with certain alloys and tempering it may develop along transgranular paths or a mixed intergranular/transgranular path.

4 Principle

4.1 Test specimens are continuously immersed in aqueous solutions of specified composition and temperature for short periods depending on the type of alloy and corrosive solution.

4.2 The relative susceptibility to exfoliation corrosion is qualitatively assessed by visual examination, with performance ratings determined by reference to standard photographs or numerical exfoliation ratings.

5 Significance and interferences

5.1 This International Standard provides a useful prediction of the exfoliation corrosion susceptibility of aluminium alloys in various types of outdoor service, particularly in marine and industrial environments. The test solutions are very aggressive and represent the more severe types of environmental service.

5.2 Interference with the realistic exfoliation ratings of certain materials can occur as a result of:

- a) powdering of the specimen surface due to severe general corrosion and the continuous dropping of extremely fine particles;
- b) the removal of exfoliation blisters or delaminations by aggressive cleaning after exposure.

6 Test specimens

6.1 Size

There is no required size or shape but it is advisable that the specimen be not too small as visual inspection is the key evaluation method. It is recommended that flat specimens at least 50 mm × 75 mm in size and full section thickness with the specimen length oriented in the direction of principal deformation of the product be used when practicable. For convenience in handling during exposure, specimens of convenient thickness may be sawn from the surface region of thick sections or bulky products.

For comparable results with a series of material variants, the size and shape should be one and the same.

6.2 Location

The test surface for semi-finished products should be either the as-fabricated surface or some specified interior planes. Interior planes typically used are:

- a) $T/10$ = 10 % of the thickness removed (this is representative of a minimal machining cut to obtain a clean flat surface);
- b) $T/4$ = quarter plane, with 25 % of the thickness removed;

c) $T/2$ = midplane, with 50 % of the thickness removed.

These interior planes are representative of many of the exposed surfaces of parts machined for aircraft components and may expose regions in the grain structure with the highest exfoliation susceptibility.

When removing test specimens from extrusions and forgings, specimen locations underneath flanges, ribs, etc. or where the grain structure is usually variable, shall be avoided.

The test surfaces should be machined and/or chemically milled to produce a uniform surface free of heat treating films, alclad coatings, uneven surface layers of recrystallized grains, traffic nicks and scratches, etc. unless it is desired to test the metal in an as-received condition.

6.3 Machining

When machining specimens for exposure of interior planes ($T/10$, $T/4$, etc.) the final machining cut shall be a light one of about 0,635 mm or less to avoid the creation of a highly worked surface (an artifact which could influence the perception of exfoliation of relatively resistant materials).

The roughness parameter of machined test surfaces, R_a shall not exceed 2,5 μm unless it is required to simulate an as-manufactured surface condition.

The test specimens shall have edges dressed by machining or filing to a depth sufficient to remove deformed metal and residual short transverse tensile stresses. If the thickness of the sheared specimen is < 3 mm the edges should be machined to a depth of 100 % of the specimen thickness, and if its thickness is ≥ 3 mm, the edges should be machined to a depth of at least 50 % of the thickness.

6.4 Alclad products

The cladding shall be removed from the test surface of specimens of alclad products and either removed or masked off on the back (non-test) surface. The cladding may be removed either by machining or chemical milling.

6.5 Weldments

For testing weldments of semi-finished products or parts the weld shall be positioned in the centre of and perpendicular to the long side of the specimen.

The length of the test specimen should be such that the distance from the outside edges of the heat-affected zones to the specimen ends is at least 30 mm.

6.6 Surface preparation

Degrease the specimen test surfaces with a suitable organic solvent and, if desired, give further cleaning by appropriate etching. A frequently used etch technique for Al-Mg series alloys is to immerse specimens for 60 s in 5 % (m/m) sodium hydroxide solution at 80 °C, rinse in water, desmut 30 s in concentrated nitric acid ($\rho = 1,4$ g/ml) at room temperature, rinse with distilled or deionized water and air dry (see ISO 11846).

6.7 Specimen identification

Because of the nature of exfoliation corrosion considerable surface metal may be destroyed along with specimen identification numbers during the exposure of susceptible materials. A permanent number can be scribed at one end of the back (non-test) surface and covered by a protective coating. A separate tag of a non-corrodible, non-conducting material is another method. Any method that deforms the material shall be avoided.

6.8 Specimen replication

Sampling procedure and the number of replicate specimens to be tested are not covered by this International Standard.

6.9 Control specimens

6.9.1 To provide an indication when some inadvertent deviation from the specified test conditions may have occurred, it is always advisable to include control specimens from known materials representing both high and low susceptibility to exfoliation. Such controls verify the validity of the particular test run.

6.9.2 The best check on the appropriateness of an accelerated corrosion test is to determine whether the results agree with known service experience. When there is no actual service experience, then exposure in a corrosive outdoor atmosphere known to produce exfoliation corrosion is a useful approximation of the conditions a part will encounter in service. The most frequently used environments are sea coast sites and highly industrialised urban locations [7–9].

7 Test solutions

7.1 It would be ideal if a single testing solution were equally effective for all aluminium alloy systems, but such is not the case with the present state of the art. Recommended test solutions for specific exfoliation – susceptible alloy systems are given in Table 1.

7.2 Testing conditions along with notations of applicable national standards and supporting published references are given in Part I of Table 2. Part II contains additional testing conditions which have shown promise for certain alloys, but with less general experience. Continued evaluation of these test solutions is needed, including comparisons with service environments.

7.3 For research studies of new types of alloys and mill products it is advisable to experiment with more than one of these test solutions, all of which have been found to have some merit and to consider a milder cyclic acidified salt-fog cabinet type of test [14, 15].

7.4 Solutions containing hexavalent chromium (Cr^{6+}) ions are not desirable in some countries due to health and environmental considerations.

7.5 All test solutions shall be prepared with reagent grade chemicals and distilled or deionized water with a conductivity not greater than $10 \mu\text{S}/\text{cm}$ (see ISO 3696).

7.6 Use a fresh solution at the start of each test.

8 Apparatus

Any suitable glass, plastic or other inert container may be used to contain the corrosive solutions and specimens during the period of test. Depending on the shape and size of the test specimens, rods or racks of glass, plastic or any inert material shall be used to support the specimens above the bottom of the container. The container shall be fitted with a loose-fitting cover to reduce evaporation.

9 Testing procedure

9.1 General procedure

Tests may be conducted in vessels of various capacities and the specimens arranged so that they are fully immersed in the test solution. More than one specimen may be immersed in the same container provided that the prescribed solution volume per area of exposed metal is maintained and specimens do not touch each other.

9.1.1 Specimens may be positioned either vertically or horizontally in the test solution. The vertical position is usually preferred for the Al-Mg and the Al-Zn-Mg series alloys which are relatively resistant to general corrosion [11]. The horizontal position is preferred for the less corrosion resistant Al-Cu and Al-Zn-Mg-Cu series alloys in solution number 2 to prevent the falling off of loosely adhering exfoliation products. For horizontally exposed specimens the test surface should be uppermost, with the bottom side being masked with a suitable protective coating.

9.1.2 Specimens of different alloy systems shall not be exposed together in the same container (e.g. alloys containing less than 0,25 % copper with those containing greater amounts of copper).

9.2 Test duration

Even though there is a prescribed test period for a given test solution, (see Table 2) it is a good practice to inspect the specimens in-situ during the course of exposure in order to note when exfoliation begins and how it progresses.

Standard tests are generally conducted for the recommended period of exposure. However, if no appreciable exfoliation laminations are observed on specimens of a new alloy or product (especially from thick sections) it may be useful to double the recommended test duration.

NOTE The length of time to develop exfoliation in material of a given alloy and temper can vary with the mill product form, with some materials developing severe exfoliation in much shorter periods than those specified.

9.3 Post-test appraisal

Carefully remove exposed specimens from the test solution at the end of the test period to prevent loose exfoliation particles floating away. Then rate their performance in accordance with Clause 11 while the specimens are still moist, taking into account all loose exfoliation products lying on the test specimen or on the bottom of the container. Photographs may be advisable at this stage.

If, in some cases, it is impossible to distinguish mild forms of exfoliation from general corrosion, it may be helpful to chemically clean the specimens by soaking them in concentrated nitric acid ($\rho = 1,4 \text{ g/ml}$) at room temperature for only a few minutes, just sufficient to dissolve corrosion products without dislodging layers or flakes of true exfoliation, followed by gentle rinsing with tap water.

NOTE Avoid prolonged soaking or scraping of the specimen test surface as this can cause the loss of exfoliation delaminations and result in erroneous performance ratings. The ratings may be too severe if general corrosion products are left on the specimen test surface, or too mild if the cleaning is too vigorous.

10 Interpretation of results

10.1 Visual exfoliation ratings

Two widely used procedures are recommended depending on the alloys being evaluated and the test solution employed (see Table 1).

Table 1 — Recommended test solutions for constant immersion exfoliation corrosion tests of susceptible aluminium alloys

Alloy Systems		Test solution ^a
ISO Designation	International registration, AA	
Al Cu6BiPb	2011	2, 5
Al Cu4SiMg	2014	2, 5
Al Cu4MgSi	2017	2, 5
Al Cu4Mg	2X24	2, 5, 6
Al Cu6Mn	2X19	2, 5, 6
Al Cu2Mg2Ni1Fe1	2618	2, 5
Al Cu2,5Li2Zr	2090	4, 6
Al Mg5Li2	1420 ^b	4,5
Al Mg5Cr	5056	3, 4
Al Mg4,5Mn0,7	5083	3, 4
Al Mg4	5086	3, 4
Al Mg5Mn1	5456	3, 4
Al Zn4Mg1,5Mn	7004	1, 2
Al Zn4,5Mg1	7020	1, 2
Al Zn4,5Mg1,5Mn	7005	1, 2
Al Zn5,5MgCu	7X75	2, 4, 6
Al Zn6CuMgZr	7X50	2, 4, 6
Al Zn6MgCu	7X10	2, 4
Al Zn7MgCu	7178	2, 4
Al Zn8MgCu	7X49	2, 4

^a See table 2 for descriptions of the test solutions.

^b According to GOST registration of alloys.

10.1.1 Procedure A

The following classifications shall be used to report the susceptibility to exfoliation corrosion of exposed test specimens by comparison with standard photographs for test solutions 2, 3 and 6:

Classification	Rating
No appreciable attack	N
Pitting	P
General corrosion	G
Exfoliation	EA, EB, EC, ED

10.1.1.1 Descriptions of the four visual corrosion classifications

- a) N: no appreciable attack - surface may be discoloured or superficially etched;
- b) P: pitting - discrete pits, sometimes with a tendency for undercutting and slight lifting of metal at the pit edges [see Figure 1a)];
- c) G: general - fairly uniform corrosion with accumulation of powdery corrosion products; the basic type of attack may be either pitting or intergranular;
- d) EA to ED: exfoliation: visible lifting of the metal surface manifested in various forms, such as blisters, slivers, flakes, fairly continuous sheets and sometimes granular particles resulting from disintegration of thin layers of metal. [see Figure 1b)] Various degrees of exfoliation with increasing area and penetration are shown in Figures 2 to 7.

NOTE The appearance of the exfoliation, which may be general (see Figures 2 to 5) or localized (see Figures 6 and 7) swellings (blisters), is influenced by the grain structure of the test material, its inherent susceptibility to general corrosion and stress corrosion cracking, and the corrosivity of the testing solution. For example, the appearance of rolled specimens of an Al-Zn-Mg-Cu alloy (7X75 type) exposed to test solution 2 (in Table 2) is illustrated in Figures 2 to 5; specimens of an Al-Mg-Mn alloy exposed to test solution 3 in Figure 6; Al-Zn-Mg-Cu specimens exposed to test solution 4 or 5 in Figure 7.

Table 2 — Exfoliation corrosion test conditions

Test solution	Concentration of components in test solution						Solution pH	Temperature °C	Solution volume ml/cm ²	Test duration ^a d	Standards (reference)
	g/l										
	K ₂ Cr ₂ O ₇	HCl	NaCl	KNO ₃	HNO ₃	AlCl ₃ ·6H ₂ O					
Part I											
1	20	9	—	—	—	—	2,5	18 to 25	10	7	Ger. TGL 18760-02; Gost 9.904-82/ (1,2,3)
2	—	—	234	50	4,4	—	0,4	25 ± 3	15	2 to 4	ASTM G34/(4-10)
3	54 g NH ₄ Cl + 20 g NH ₄ NO ₃ + 2 g (NH ₄) ₂ C ₄ H ₂ O + 3 g H ₂ O ₂						5,2/5,4	65 ± 1	10	1	ASTM G66/(6, 11)
4	20	13,5	—	—	—	—	1,0	18 to 25	10	7	GOST 9.904-82; (1, 2, 10, 12)
Part II^b											
5	10	—	234	50	4,4	—	0,8	25 ± 3	15	2 à 4	GOST 9.904-82; (1, 2, 10, 12)
6 ^c	—	—	234	61	—	5,4	3,2	25 ± 3	20	4	(13)
^a For research purposes it is permissible to increase the test durations to as much as double the recommended periods so long as the general corrosion does not interfere with assessment of exfoliation. ^b Solutions 5 and 6 are corrosion inhibited versions of solution 2 (ASTM G 34) to minimize general corrosion to facilitate the assessment of exfoliation. ^c Solution 6 is used at 25 °C ± 3 °C for Al-Cu alloys and at 52 °C ± 3 °C for Al-Zn-Mg-Cu and Al-Cu-Li alloys.											

10.1.1.2 Rating guidelines

The visual exfoliation ratings are intended to be finite indications of the resistance to exfoliation of the test material, and care should be taken when rating a series of material variants to compare each with the photographs in Figures 2 to 7, rather than with each other.

10.1.1.2.1 When the appearance of a test specimen appears to be borderline between adjacent exfoliation ratings, choose the rating indicating higher susceptibility.

10.1.1.2.2 When exfoliation occurs at localised sites, rate the worst localized condition observed.

10.1.1.2.3 When the presence of exfoliation is questionable because of large amounts of powdery corrosion products, metallographic examination of a cross-section of the corrosion will be required to determine the correct rating (see Figure 1).

10.1.2 Procedure B

This procedure involves a numerical rating of the degree of corrosion damage on a scale of 1 to 10 using the following visual criteria for test solutions 1, 4 and 5:

- Percentage of a test surface area that is exfoliated;
- Change in specimen appearance, including extent of edge cracking;
- Diameter, in millimetres, of the largest blister or area of exfoliation.

Details of the procedure are given in Table 3. See examples in Figure 7.

10.2 Quantitative exfoliation ratings

The lack of a generally accepted numerical measure of the corrosion damage due to exfoliation hampers analysis of test results when a number of material properties are considered for a combination ranking of several candidate materials of construction. There is insufficient information available for standardization, and when a numerical criterion is required, some procedure should be agreed upon by the concerned parties (see annex A).

11 Factors that affect reproducibility

11.1 The major factors affecting reproducibility of the test results for a given composition of test solution are:

- a) Solution temperature;
- b) Ratio of volume of solution to area of metal exposed;
- c) Duration of test;
- d) The subjectivity of the visual rating systems;
- e) Variability in the test material.

When the testing conditions are controlled and the test material is uniform, the main factor would be the relatively subjective visual rating systems. Considerable American experience with the EXCO test (ASTM G34) and the ASSET test (ASTM G66) has shown under controlled test conditions that a rating difference of one letter grade is not uncommon when a set of test specimens is rated by various inspectors ^[14].

11.2 The procedures in this test method have no bias because the results are defined only in terms of the test method, and there is no absolute standard for reference. However, this method does rank materials according to their performance in marine atmospheres [6, 8, 9, 10, 13].

12 Test report

The test report shall contain the following information.

- a) Alloy, temper designation and chemical composition of test specimens;
- b) Type of semi-finished mill product, or part, and section thickness;
- c) Applicable product specification;
- d) Sampling procedure and number of replicate specimens;
- e) Specimen location, size and surface preparation;
- f) Test solution and period of exposure;
- g) Any deviation in test procedure from that described in this International Standard;
- h) Ratings of the test specimens according to clause 10 of this International Standard.

Table 3 — Numerical exfoliation ratings

(adapted from GOST standard 9.904-82)

Rating number	Exfoliated area %	Change in appearance diameter of blisters mm	Total length of edgecracking %
1	0	N (see 10.1.1)	0
2	0	P or G (see 10.1.1)	0
3A ^a	up to 2	1 max.	0
4A ^a	2 to 5	3 max.	0
5B ^a	5 to 10	5 max.	0
6B ^a	10 to 25	> 5	up to 10
7C ^a	25 to 50	> 5	10 to 25
8C ^a	50 to 75	Thickening up to 10 %	25 to 50
9D ^a	up to 100	Thickening 10 % to 25 %	> 50
10D ^a	up to 100	Thickening > 25 %	> 50

NOTE 1 The exfoliated area of each specimen is estimated visually and expressed as a percentage of the test surface area excluding edges. A transparent grid with squares of 1 mm to 5 mm per side may be used for this.

NOTE 2 The exfoliated surface area will generally be significantly larger for copper-bearing alloy specimens exposed to uninhibited test media such as solution number 2 (Table 2): e.g. compare the photographs in Figures 2 to 5 with those in Figures 6 and 7.

NOTE 3 If the exfoliated surface area corresponds to a small rating number X, such as 3, 4 or 5, but the maximum diameter of individual blisters corresponds to a higher rating number, then the specimen should be rated at X + 1 (i.e. 4, 5 or 6).

NOTE 4 If the exfoliated surface area corresponds to an intermediate rating number X, such as 5, 6 or 7, and the diameter of individual blisters does not exceed 5 mm, then the specimen should be rated X – 1 (i.e. 4, 5 or 6).

NOTE 5 To evaluate the change in appearance of a test specimen it is necessary to note the presence of pits, general corrosion, the maximum diameter of exfoliation blisters, and any thickening due to delaminations.

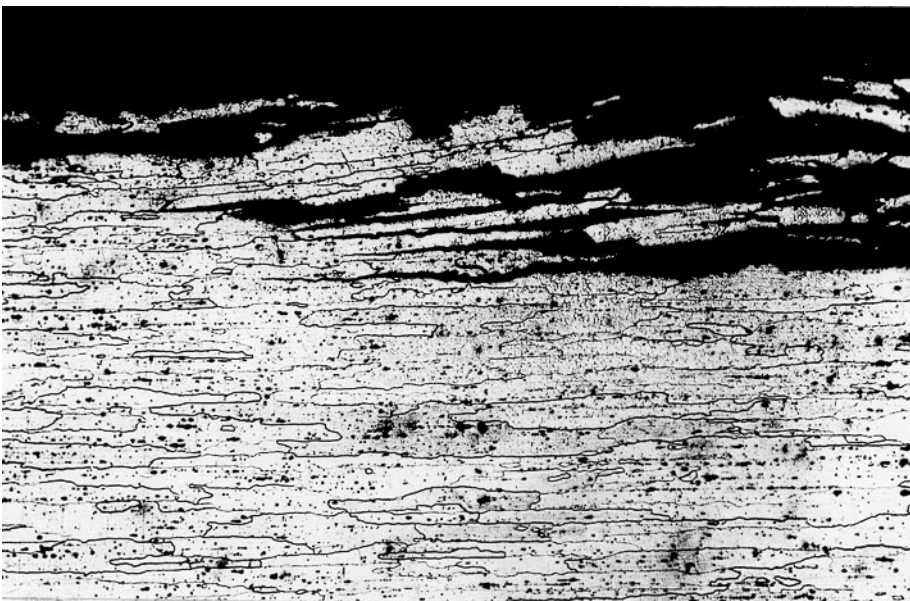
NOTE 6 The final exfoliation rating of a specimen is determined by the adjusted rating number for the percentage exfoliated area.

NOTE 7 The test material is evaluated by calculating the arithmetic average rating for the replicate specimens.

^a Approximate exfoliation rating in accordance with 10.1.1 (ASTM G34).



a) Undermining pitting that may from the surface give the appearance of incipient exfoliation.



b) Exfoliation resulting from rapid lateral attack of selective boundaries or strata forming wedges of corrosion product that force layers of metal upward giving rise to a layered appearance.

Figure 1 — Metallographic sections illustrating two different types of attack (Keller's reagent) 100 ×

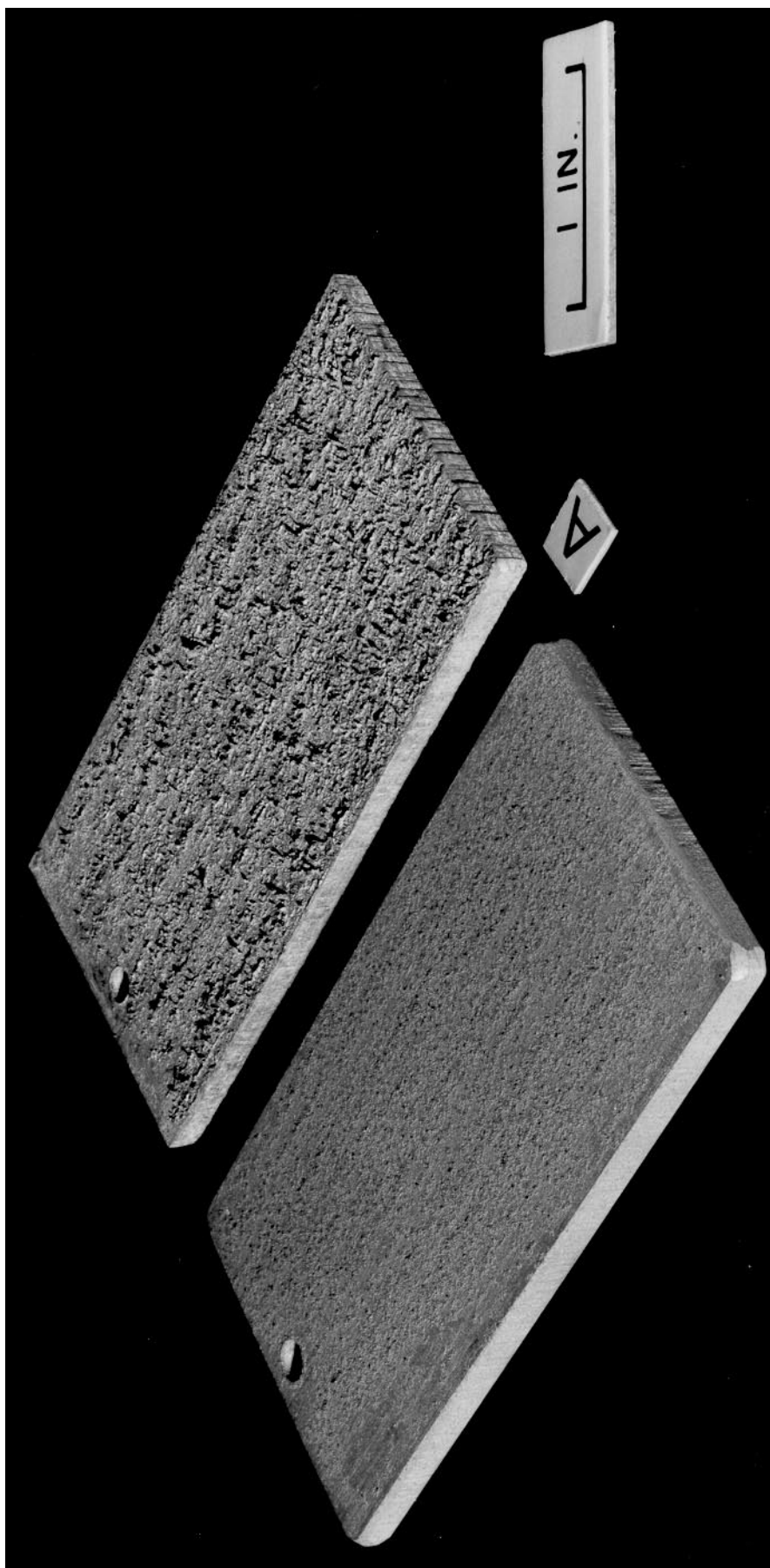


Figure 2 — Examples of exfoliation rating EA (superficial): thin slivers and flakes with negligible penetration into the metal; could be in the form of localized tiny blisters up to 1 mm maximum diameter. (See also Figure 6 and photos 3 and 4 in Figure 7)

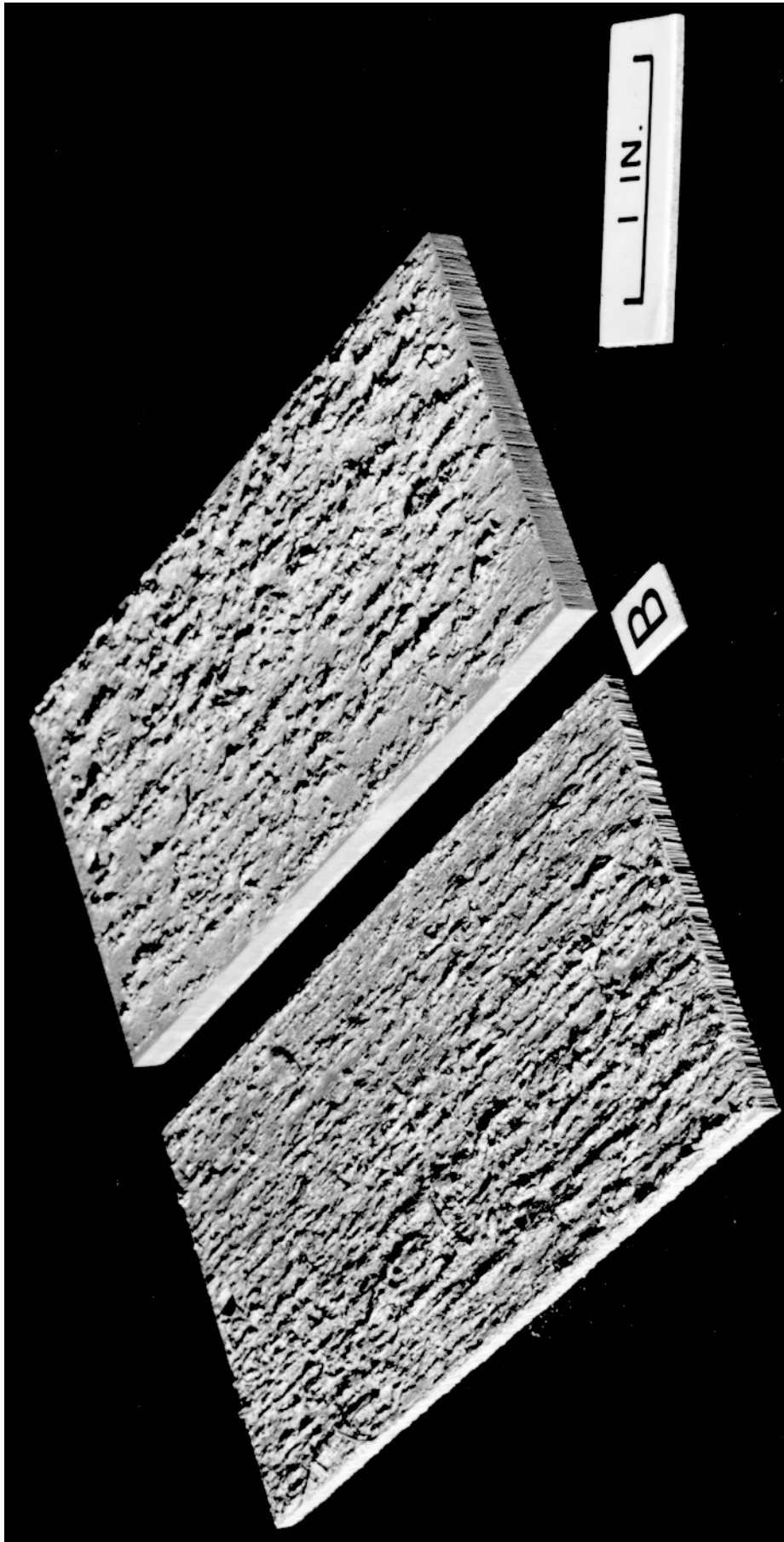


Figure 3 — Examples of exfoliation rating EB (moderate): general blistering and layering but with only slight penetration into the metal; could be in the form of localized blisters up to 5 mm maximum diameter. (See also Figure 6 and photos 5 and 6 in Figure 7)

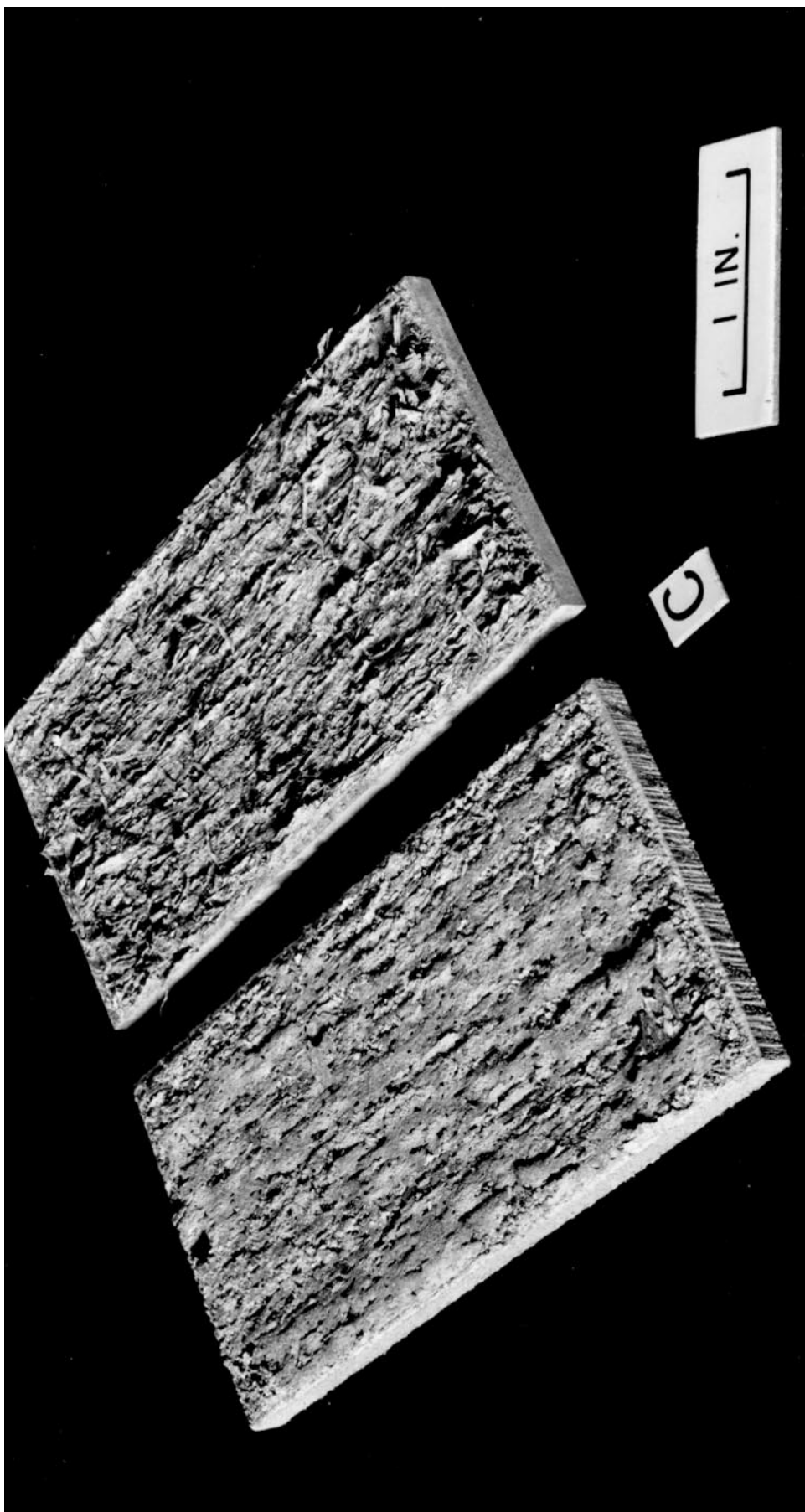


Figure 4 — Examples of exfoliation rating EC (severe): general blistering and delamination with penetration up to approximately 10 % of the specimen thickness. (See also Figure 6 and photo 8 in Figure 7)

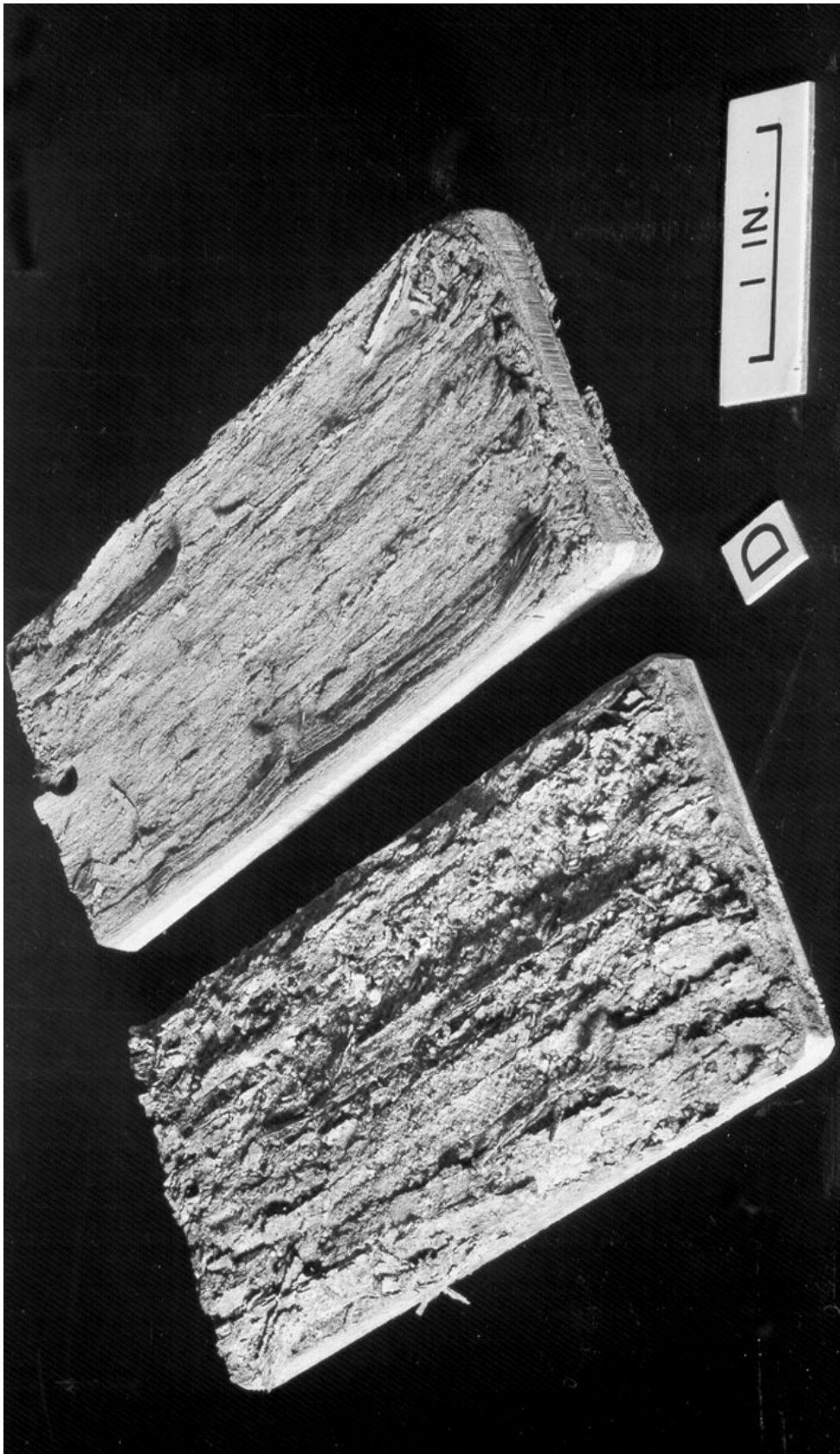


Figure 5 — Examples of exfoliation rating ED (very severe): general delamination involving over 10 % of the specimen thickness. (See also Figure 6 and photos 9 and 10 in Figure 7)

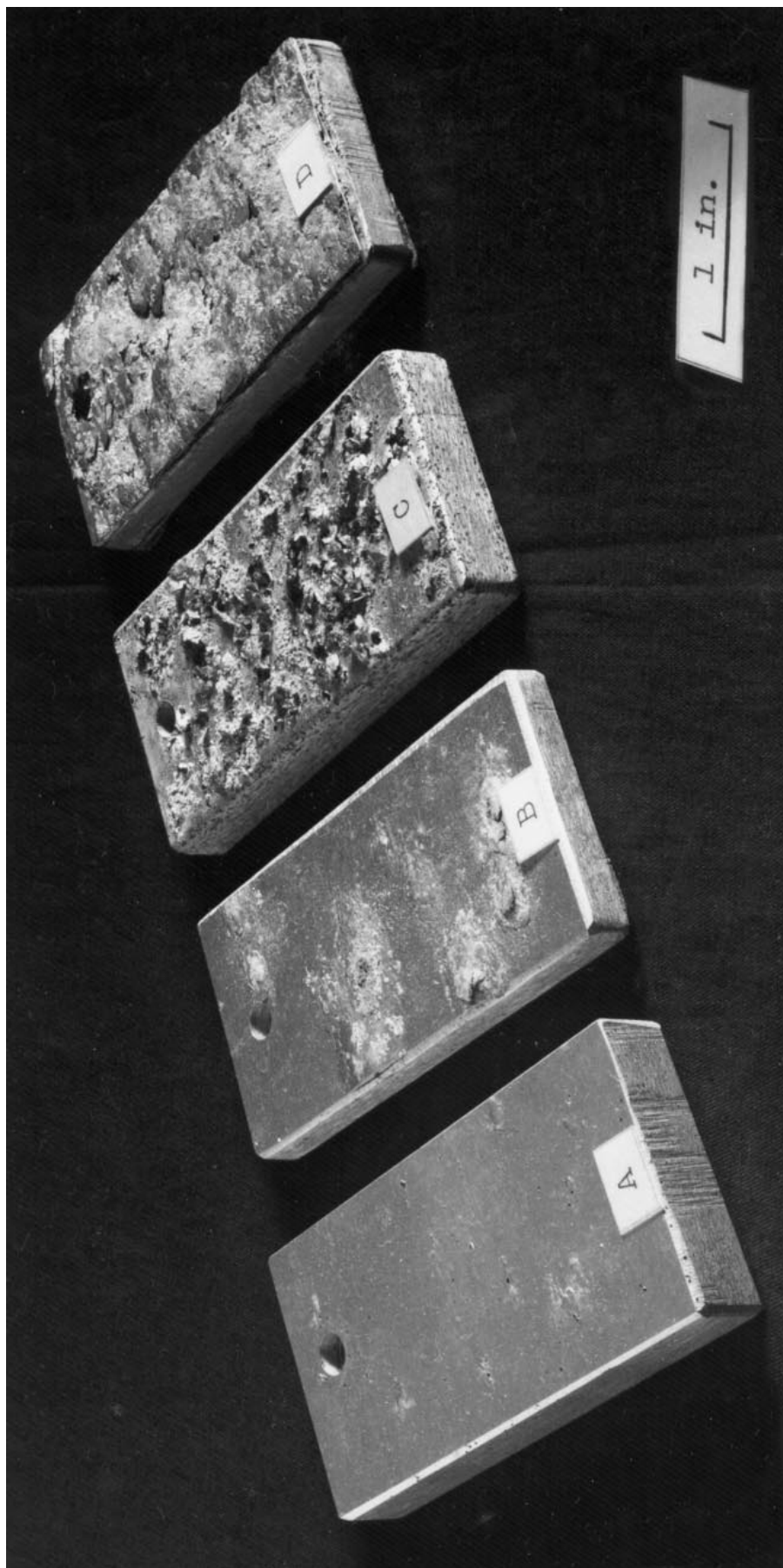
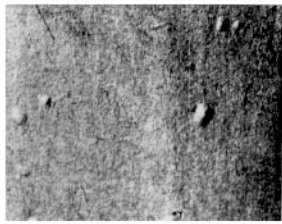


Figure 6 — Examples of four degrees of exfoliation (ratings EA to ED) that originated in form of localized blisters. (See also Figure 7)



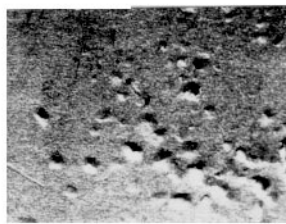
Number 3



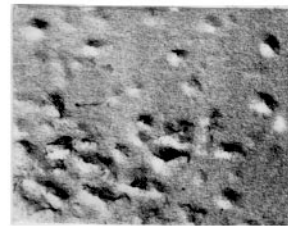
Number 4



Number 5

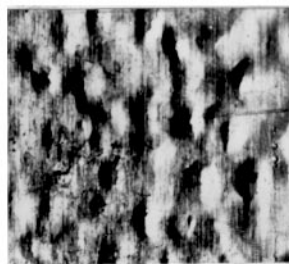


Number 6

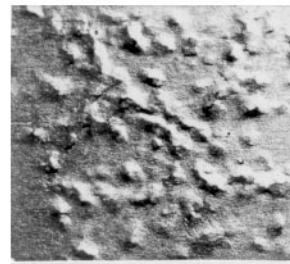


Number 7

25 mm



Number 8



25 mm



Number 9



Number 10

10 mm

Figure 7 — Typical examples of exfoliation corrosion ratings of Al-Zn-Mg-Cu alloys in solutions 1, 4 or 5; view from above and side view

Annex A

(informative)

Quantative exfoliation rating

Promise for less subjective numerical ratings has been shown in recent investigations, but the procedures have not yet been generally accepted for standardization:

- a) measurement of pressure build-up during exfoliation ^[16];
- b) measurement of depth of corrosion using an ultrasonic probe ^[17].

Another approach is to assign numbers as substitutes for the letters. It is proposed for this purpose that a geometric scale (such as EA = 1, EB = 2, EC = 4, ED = 8) would be consistent with the increasing corrosion damage shown by the photographs in Figures 2 to 7.

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