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CORROSION TESTING FOR METAL FINISHING

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(editor)

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Preface

Metal finishing has two basic aims, firstly, to treat a metal to obtain a high degree of acceptable appearance and, secondly, to protect the finished article from deterioration. Individual applications may fall into either category or involve combination of both aims. It follows, therefore, that metal finishers will be closely concerned with performance and methods of testing for corrosion resistance both from the aspect of choosing the correct finish for the application and for quality control checks on production.

There is a wide range of corrosion test methods available, the majority being the subject of Standard Specifications or currently being brought to Standards status. These Standards set out in detail the apparatus required, the procedure to be adopted and also indicate the principal finishes for which their use is intended.

This book presents to the metal finisher the whole range of these test methods and gives guidance in the choice of method for a particular finish. With many product Standards there is a choice of test methods available for meeting the Standard requirements, with others test methods are not specified but the producer and/or user may need to carry out some form of corrosion testing. It is hoped that the relevant choice may be obtained more easily as a result of the information published here.

The apparatus and procedure for each test method is outlined and its applicability to different metals and finishes is discussed. Indications are given of the nature and extent of the corrosion which develops in the test.

Reference is made to the relevant Standards for each test method and *it is of the first importance that the user should obtain the relevant Standard document* when he has chosen the test method to be used for his particular application so as to ensure that the test will be operated in strict conformity with the requirements of the Standard so that results can be compared with those obtained by others using the same test for the same type of product.

V. E. Carter

Sadly, Mr V. E. Carter died soon after completion of this book. The Institute of Metal Finishing and the publishers express their gratitude to Dr Michael Clarke of the City of London Polytechnic for reading the proofs.

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Preparation for testing

When considering corrosion testing of finished metal products there are three basic requirements:

- (1) The type of test specimen.
- (2) The method of preparation for corrosion testing.
- (3) The choice of corrosion test method.

Variations within this group of requirements can have considerable effect on the significance of the results achieved.

Type of test specimen

Test specimens can be in the form of specially prepared flat panels or, alternatively, the tests can be carried out on production articles. Tests using flat panels are of greatest use for investigating the basic corrosion behaviour of particular finishing systems and have the added advantage that because of their regular area and flat shape assessment of corrosion damage is most easily achieved. With a flat test panel the thickness and uniformity of a coating system is more easily obtained and maintained. In addition the whole of the test surface can be exposed to the corrosive environment to a uniform degree. Taken together these factors ensure that the corrosion pattern remains as uniform as possible over the greater part of the test surface – provided that a sufficient margin around the sides of the panel is ignored during assessment so as to eliminate edge effects.

The disadvantage of using flat panels lies in the fact that the application of the finishing process to production articles rarely results in complete uniformity of finish. Electroplated coatings vary in thickness with the geometry of the shaped surface and with the throwing power of the plating process used; with hot-dipped metal coatings and painted coatings thickness is influenced by liquid run-off from edges and its retention in recesses, and with sprayed finishes thin areas may also result from shielding from the

2 Preparation for testing

spray as a result of complexities of shape. Additionally, any grinding or polishing done during finishing may produce differing degrees of work-hardening dependent upon geometry which could influence service performance. The effect of all these variables is to change the pattern of corrosion on the article as a whole with some areas remaining free from breakdown while others suffer enhanced attack. The effect is compounded by the unevenness of exposure to the corrosive environment – i.e. recessed areas may retain greater amounts of corrodant for longer periods whereas corrodant runs off more readily from sharply angled areas. Consequently, when using corrosion tests for quality control acceptance purposes or as a means of determining performance for particular components, it is always preferable to test actual production articles.

In the case of paint coatings it is necessary to use specially prepared test panels when the purpose of the test is to determine the basic properties of the paint film *per se* (e.g. loss of gloss or colour, chalking, checking, cracking or crazing) rather than its ability to protect a given substrate. Standards for paint testing usually specify details for the preparation of these special test panels. If, however, the purpose of the test is to determine performance of the paint coating in respect of its ability to protect a given substrate, either flat panels or production articles may be used as appropriate to the exact nature of the information required.

When using production articles for corrosion testing it may be necessary to increase the replication so that several different orientations of the test specimens to the corrosion environments can be achieved, thus ensuring an even degree of exposure of all significant surfaces or so as to achieve any specific 'corrosion traps' which *might* occur in service. The assessment of corrosion on production articles is more difficult than on flat panels because of the problem of accurate determination on surface area and also the need to relate the different patterns of corrosion to specific areas when reporting results.

Method of preparation for corrosion testing

The method of preparing specimens for corrosion testing can markedly affect the results achieved. It is, of course, necessary to remove any extraneous soils or greases from the finished surface so as to allow free access of the corrodant (unless the use of a temporary protective such as grease or lacquer would form part of

the service usage). Removal of soils or greases must be done in such a way that the normal surface condition of the finish remains unaltered. Some cleaning or degreasing agents can themselves react with the finished surfaces or alter naturally formed films on these surfaces. Consequently, it is necessary to make a careful choice so as to avoid changes which might affect corrosion performance.

Similarly, the way in which the cleaning agent is applied can affect the efficacy and/or the chances of causing surface reactions. Thus, simple immersion in a cleaning solution may not remove oils or greases completely but may leave a very thin film on the finished surface which will reduce or prevent subsequent corrosion. A useful guide to the efficacy of removal of contamination is to check whether a 'water-break free' surface has been obtained – if not, the presence of remaining grease films is likely. Brush or swab application of cleaners may damage or detach poorly adherent oxide films from the finished surface and the use of hot cleaning solutions or vapours may strengthen naturally formed oxide films.

Particular care is necessary when preparing painted surfaces or metal surfaces which have received chemical passivation treatments and in some cases cleaning procedures should be omitted before corrosion testing.

Guidance on the use of suitable procedures for preparation for specific corrosion tests is usually included either in the Test Method Standard or in the individual Product Standard and the specific method should be used unless special considerations apply. It is always desirable to record the method of preparation in the report on the corrosion test results so that any differences between results from separate testing organizations can be identified. In cases where there are doubts as to whether the method of preparation has affected the results replicate tests with different preparation procedures while retaining all other test parameters constant may be desirable.

The choice of corrosion test method

The choice of corrosion test method to be used will depend upon the following factors:

- (1) The information required.
- (2) The nature of the article to be tested.
- (3) The particular finish to be tested.
- (4) The intended end use of the article.

The information required

(a) *Establishing service performance* The only wholly reliable tests for establishing service performance are actual exposure to the natural environment encountered in service. It is necessary to select the type of environment (e.g. industrial, rural, marine), the type of exposure (e.g. static or mobile) and the orientation of the test specimens so as to achieve the best reproduction of the service usage. The major disadvantage is the length of testing time required to obtain useful data so that this method of testing is normally chosen only for research and development purposes, for obtaining factual data on which design or warranty considerations can be based or for cases where it is essential that good knowledge of likely service performance is essential – e.g. applications involving major safety considerations. It must always be borne in mind that the results of exposure to natural environments may vary with climatic changes from season to season or from any one season or year to a subsequent season or year.

(b) *Reproducing service behaviour* To obtain data more rapidly than by exposure to natural environments a compromise may be sought by using accelerated tests specifically designed to reproduce similar patterns of corrosion to those encountered in service within a limited test period (generally several hours). It is essential when using such tests that comparison data for test results with those of service exposure be obtained using materials of known performance so that a correlation can be established to allow interpretation from the tests to service to be made with at least some degree of confidence. At best – i.e. when good correlation has been achieved – some indications of the rate and pattern of breakdown in service may be obtained, but at worst the test will provide only a rough order of merit for different groups of specimens compared with ‘standard’ specimens tested at the same time. Examples of these types of test are the CASS test comparing with outdoor service of electroplated coatings cathodic to the substrate and the Corrodokote test for reproducing the effect of road-wash deposits on electroplated coatings cathodic to the substrate which are used in motor vehicles.

(c) *Quality control* For quality control purposes it is not essential to reproduce the pattern or extent of service breakdown in the accelerated test. The test must differentiate between the performance of an article which has a quality giving adequate service performance and one whose quality is inadequate; reproducibility in the test must be good and the test period short enough to allow remedial measures to be taken to improve the standard of

production when loss of quality is detected. For any given type of finish it should be possible to select one or more accelerated tests suitable for quality control purposes (see *Table 1* and details in the individual test specifications in the remaining chapters) and in many cases guidance on particular test methods to be used for quality control purposes is given in individual Product Standards.

TABLE 1. Suitability of corrosion test methods to individual finishes

<i>Test method</i>	<i>Anodic oxide</i>	<i>Aluminium</i>	<i>Copper</i>	<i>Zinc</i>	<i>Cadmium</i>	<i>Nickel and chromium</i>	<i>Lead</i>	<i>Tin</i>	<i>Stainless iron or steel</i>	<i>Precious metals</i>	<i>Passivated</i>	<i>Organic</i>	<i>Refer to chapter</i>
Outdoor	X	X	X	X	X	X	X	X	X	X	X	X	5
Humidity		X	X	X	X							X	7
Salt droplet			X	X								X	8
Neutral salt	(X)	X	X	X	X	X	X		X			X	
ASS	X	X		X	X	X	X		X			X	
CASS	X					X			X			X	
CRL beaker												X	9
Kesternich	X					(X)			(X)			X	
AT & E										X			
Clarke and Leeds										X			
Industrial													
atmosphere										X			10
Thioacetamide										X			9
Preece				X				X					11
Ammonium								X					
persulphate								X					
Ferroxyl						X	X	X		X			
Electrographic						X	X	X		X			
Dubpernell						X							
Gassing				X	X								
Corrodkote						X			(X)				12
EC test						X							13
Alternate													
immersion		X				X						X	14
Bimetallic	X											X	15
Mortar	X											X	16
Acid													
immersion	X												17
Dye spot	X												
Admittance	X												
Artificial													
weathering													
Light-fastness	X											X	18
Anti-perspiration			X	X	X	X				X	X	X	19
													20

Entries in parentheses indicate poor discrimination.

A further consideration specific to quality control testing is the standard of acceptance level to be set when considering the results. Ideally, quality control tests should be of a non-destructive nature so that production items are not destroyed. This may be difficult or impossible to achieve but some 'indirect corrosion tests' such as thickness measurement by eddy current and sealing tests for anodized aluminium such as admittance and dye-spot tests are wholly non-destructive. Some porosity tests using less severe corrosive environments which only reveal existing pores rather than breaking down weak points in a coating may be considered as virtually non-destructive to good quality production where the only consideration is the criterion of complete coverage of the substrate. In all other cases the test is of necessity destructive and an acceptable level of corrosion must be established. This may be on a 'go-no go' basis but the dangers of failing to detect reduced severity of test (and hence the acceptance of poorer quality) make it more desirable to use a test which will produce some breakdown even on materials of acceptable quality and to set an arbitrary level of corrosion beyond which the material is considered to be of unacceptable quality. For this purpose a suitable assessment method for deterioration in the test must be used (see chapter 4) and the individual acceptance levels to be used for a given method and finish may be found either in the Test Method Standard or, more usually, in the relevant Product Standard.

The nature of the test article

The nature of the test article must be taken into consideration when selecting a test method. Clearly, if an article is too large to be accommodated in a test cabinet it cannot be tested complete and cutting it in pieces may introduce defects which will affect the validity of the test. Conversely, the article may be too small to be handled or exposed in the specified manner - e.g. the surface area is too small to apply an electronic probe head.

Flat panels are easily exposed at any given orientation - e.g. to allow free settlement of a salt fog corrodant on the surface - and they readily allow the application of an electrographic porosity test paper whereas shaped articles will require many replicates to expose all areas to a given environment or it may even be impossible to wholly meet the requirements of a given test. Thus for shaped articles gaseous or immersion porosity tests must be used. Complicated shapes, particularly those with deeply recessed and re-entrant angled areas, as well as very small components may be impossible to coat with the Corrodokote slurry or receive complete

exposure to salt fogs in which case alternative test methods may be required.

The particular finish to be tested

The applicability of individual test methods included here for particular finishes is indicated in *Table 1*. Selection of the best methods indicated as suitable in the table for any particular application will depend upon the type of information required as well as the article to be tested (as discussed above) and also the varying degrees of response of the particular finish to each different test. Further guidance on the final choice to be made will be obtained by studying the particular chapters concerned and also the requirements of the relevant Standards.

The intended end use of the article

The choice of test method may be influenced by the intended end use of the article to be tested. Thus, it would probably be less suitable to choose a test involving exposure to sulphur gases when the article will be used in a marine environment – a salt fog test would be better. For articles intended for use in automobile service the best choices are probably the CASS or Corrodokote tests specifically designed to reproduce the corrosive effects of exposure to road wash; also when using outdoor exposure tests for these applications mobile exposures should be included in the test programme. Outdoor exposures of materials for architectural applications should be in the type of environment most nearly representative of the service requirement – e.g. industrial, urban, rural or marine.

Quality control testing of coatings using accelerated corrosion tests

Protective coatings most commonly applied to metals to preserve appearance and function, fall generally into five categories:

- (A) Anodic (sacrificial) metal coatings such as zinc or cadmium on steel.
- (B) Cathodic (corrosion-resistant) metal coatings such as nickel plus chromium.
- (C) Anodic oxide coatings on aluminium.
- (D) Organic (e.g. paint) coatings.
- (E) Chemical conversion coatings.

To satisfy the interests of both user and manufacturer Standards specifying coating thickness *vis à vis* environmental service conditions are used to form the basis of the quality of supply. Standards in common use for the different categories are: (A) BS 1706 (ASTM A164/5-55 and ISO 2085/2), BS 2569 and BS 3382¹⁻³; (B) BS 1224 (ISO 1456/7)⁴; (C) BS 1615 and BS 3987^{5,6}; (D) BS 5493, BS 4842 and BS 3900⁷⁻⁹; (E) BS 3289, ASTM B201-68 and DEF STAN 03-11^{10,11,12}. They normally stipulate a minimum thickness of coating required for a particular service condition and in certain cases (notably threaded components) maximum coating thickness may also be specified. Determination of minimum coating thickness is a basic requirement of quality control of coated articles conforming to specifications; however, thickness determinations alone do not fully describe coating quality. For anodic oxide coatings on aluminium, however, quality control is principally achieved by means of tests of thickness and of quality of sealing (see chapter 17).

There are few items processed in the metal finishing industry that can be simply assessed for quality by determination of minimum coating thickness. Shape alone can prevent routine thickness determinations being made in recesses or other inaccessible areas. Individually processed articles with significant surfaces of substantial area and simple configuration may well be

readily controlled by carefully located thickness determinations but with small articles of complex shape (e.g. threaded components) which may be frequently treated in bulk (as in barrel plating) thickness can only be controlled by defining average batch requirements – i.e. the total coating weight expressed as an average thickness over the whole of the coated surface. In such cases individual items in any batch may have coating thicknesses which will be grossly inadequate for the service condition.

In order, therefore, to reinforce the degree of quality control obtained by determining thickness, accelerated corrosion tests on random samples from production runs are frequently specific and widely used by large manufacturing and purchasing companies. By these means defective or weak areas of coatings on significant surfaces which could be missed on inspection or thickness checks will be revealed by their inability to withstand the specified corrosion test conditions chosen to be suitable for a given quality of coating.

The accelerated corrosion tests most widely used in quality control testing of the various categories of coatings are: (A) neutral salt spray test (*see* chapter 8); (B) ASS and CASS tests (*see* chapter 8); (C) CASS and sealing tests (*see* chapters 8 and 17); (D) humidity, sulphur dioxide and weatherometer tests (*see* chapters 7, 9 and 18); (E) humidity tests.

Finally, although accelerated corrosion tests are an essential reinforcement of quality control procedures and give results reproducible within the limits of their application as specified, their use to predict environmental performance should generally be avoided since the degree of correlation which can be achieved is frequently not known and even when correlation is available it is usually only of very limited degree.

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