

A PROCEDURE FOR TESTING THE EFFECT
OF VAPOR PHASE CORROSION
INHIBITORS ON COMBINED MULTIMETALS

By

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ABSTRACT

This study presents a quick and easy to implement Vapor Phase Corrosion Inhibitor (VCI) testing procedure which is capable of validating the protective effects of VCI foils in connection with various, electrically conductive, connected metals. Furthermore it may be used to demonstrate the effects of VCI for potential customers. The test procedure demonstrates various approaches as to how deficiencies in existing test procedures can be avoided with regards to the practice orientation - the temporary corrosion protection of machines. It also points out the lack of alignment of existing test procedures to meet the demands of new VCI recipes. The considerations made during the development of the test procedure are explained in this study with regards to their effects on the VCI protective influence.

Keywords: VCI, testing procedure, combined metals

INTRODUCTION

As a result of the increased specialization of globally distributed manufacturers, the world-wide transport volume and therefore the value of the transported goods are increasing continually. In cases of the delivery of goods which, as a result of damage, are

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unusable, just-in-time concepts, maintained by the recipients of the goods, quickly allow not only the costs for the replacement, but also, in some cases, the costs for the loss of production to go up by millions. It is for this reason that, parallel to the growing transport volume, in particular the importance of the protection of blank, metallic goods is growing. In this context, the temporary corrosion protection is a form of protection for which VCI (volatile corrosion inhibitor) has enjoyed increasing popularity in the past years. More and more firms are becoming aware of this form of corrosion protection and are investigating the question regarding whether and which VCI can be used for the protection of their products during transport.

Due to the fact that, even after approximately 40 years since the introduction of the VCI by the firm Shell, the exact way in which VCI works is not known, one is strongly dependent on the testing results on defined metal samples in order to gain information regarding the effectiveness and possibilities for use of the VCI recipes.

However, should it be a question of conclusions regarding the usability on industrial components and products, then these testing results generally are not very meaningful, due to the fact that the majority of the testing procedures employed today were developed for a generation of VCI which has now almost completely disappeared from the market. Dicyclohexylammonium nitrite, for example, used to be the most frequently used VCI for the protection of iron and steel. However, since the claim has been made that this VCI, which is extremely reliable in its protection, constitutes a health risk as a result of its nitrite contents, it has been used less and less frequently. In addition to the aspects of health, the requirements of the market also change. The importance of corrosion protection for pure iron products has decreased, due to the fact that the use of non-ferrous metals has increased and the effects of dicyclohexylammonium nitrite, in some cases, even promoted the corrosion of these products [1].

The VCI testing procedures only did justice to this gradual substitution of the VCI recipes to a certain extent. Therefore, faults can be found in very many of the currently existing, standardized testing procedures, due to the fact that they do not do justice to the new circumstances in the following points:

- At the same time as the new chemical compositions, the physical characteristics also changed, i.e. the steam pressure, which, in the new VCI-dispensing, can be lower by several ten percentages in comparison to the dicyclohexylammonium nitrite. This means that the quantity of the VCI sample is insufficient in connection with the duration of conditioning for the old testing procedures - the VCI protection is not built up completely before the corrosive action.
- More recent VCI-recipes are now also capable of protecting iron and NE-metals at the same time, however, almost exclusively steel samples are used as metal examination

subjects. It is also rare that other metals are used as samples, but practically never in simultaneous combination.

- Unlike previously, the VCI are now no longer exclusively on offer in the form of powder or attached to paper. The current supply forms have become considerably more comprehensive (i.e. VCI foam, tablets, felt). However, the test apparatus generally only allow for very few supply forms such as paper and foil.

In addition to this, in the majority of cases, the practical circumstances, such as:

- The VCI packaging (protective effects only over the steam phase and not over the direct contact) and
- The corrosive action (not a one-off and, above all, no sudden - but intensive - condensation of the metals in practice) are not taken into consideration.

All of the deficiencies described demonstrate clearly that it is out of the question to speak of a direct link to practical experience with the variety of the existing, standardized VCI testing procedures. Therefore, in the following section, a new testing procedure will be presented, which, to a greater extent, does justice to the practical requirements.

EXPERIMENTAL

In corrosion tests, the corrosive action is usually generated with the aid of the formation of condensed water on the metallic test subject. This is achieved using a difference in temperature between the 'cold' metallic test subject and the 'warm' and moist air. The temperature difference can either be forced by cooling the metallic test subject or by heating the surrounding air. The extent of the temperature difference must at least be large enough so as to drop below the dew point in the prevalent relative humidity, as no condensation of the metal sample would otherwise occur. The temperature difference is either maintained or is allowed to disappear by leaving the test apparatus alone.

The corrosive action can be heightened by the addition of corrosive gasses. This was not the case in the described test procedure, due to the fact that it cannot be excluded that the corrosive gasses react chemically with the VCI and therefore suffer disturbance of their protective effects.

MATERIALS

The selected sample for the testing procedure presented here was a VCI foil of the dimensions 45 cm x 80 cm. During the course of the test, this is made into a test envelope. In this way, on the one hand, a typical case of use, as found under practical circumstances, is recreated. On the other hand, it is avoided that, if the envelopes are used several times, such as glass, VCI molecules, sticking to the envelope, falsify the test results as a result of insufficient cleaning. The size of the foil, in a proportionate relationship to the enclosed volume, is oversized, in comparison with typical details from manufacturers

(approximately 24 m²/m³ air). The oversizing serves to achieve that even deviating manufacturers' details are not fallen short of. Additionally, 5 strips of the VCI foil are needed, measuring 2 x 12 cm, with the aid of which the contact effects of the foil are examined.

In the determination of the test material, the metal sample was first selected. In order to do justice to modern machine engineering, the test should validate simultaneously the VCI protective effects on several metals, connected conductively with each other. This is of particular importance in view of the fact that corrosive procedures may take a different course as a result of the contact between several different metals (galvanic corrosion) in comparison with metals which are tested individually. The various metals should be materials which are used frequently. Five different metals were selected - St 37 (DIN EN 10025; EU 25-72-FE 360 B), rust-resistant, austenitic stainless steel, brass, aluminum, galvanized sheet steel - which were connected to each other conductively over a copper rail. The metals are used as metal sheets with the dimensions 50 x 75 x 3 mm and with a 5 mm drilled hole, centered in the proximity of the edge of the short side (diagram 1). The proportionate relationship between the mass and the surface of the metal samples has direct effects on the temperature difference between the sample and the surroundings and therefore on the corrosive action caused by the condensed water. An high sample mass means also an high thermal capacity; a large surface means a speedy evening out of temperature between the surroundings and the metal sample. For example, in this context, a relatively light but compact metal sample can be subjected to a longer duration of condensation under constant temperature burdening than an heavy, but thin sheet of metal. The phase of condensation is relatively short as a result of the proportions selected here, due to the fact that the metal samples adjust to the surrounding temperatures relatively quickly.

In addition to the short duration of condensation, the large surface area of the metal samples offers the advantage that the corrosion effects are easily recognizable. Additionally, the surface area of each metal sample can be divided into two sections. The first section is for the examination of the VCI protective effects over the gas phase and the other section is for the examination of the contact effects. The sizes of the metal sheets also makes it easier to handle them during the test preparation.

A board of synthetic material with the dimensions 150 x 180 x 2 is required as an aid. This serves to separate the metal examination subject from the water collecting on the base of which collects here to flow off. Two plastic sheets, bent to form a semi-circle, are attached to the synthetic material board. On the one hand, these serve to elevate the synthetic material board approximately 2 cm off the ground and, on the other hand, keep a distance between the test envelope and the metal examination subjects. Both the synthetic material used and the connections between the plastic sheets and the board must be resistant up to a minimum temperature of 80° C.

In addition to the objects which have already been described, the following are also required:

- Over with a temperature regulation up to 80°C
- Installation for the welding together of foils
- Sanding plate and sandpaper with 320er graining
- Methanol and fluff-free cleaning towels
- PE-foil, 100 µm thick
- Synthetic nuts and bolts, size M4
- Paper towels, approximately 12 x 12 cm
- Deionised water
- Adhesive tape

PREPARATIONS

In order to carry out the test, one control sample and two VCI samples are required. In order to prepare the metal samples and with the exception of the galvanized steel sheets, they are sanded down on the sanding plate with 320er graining under running tap water on both sides. In this context, care must be taken that no two different metals are treated with the same sandpaper. Subsequently, they are dried down with a paper towel. An additional cleaning of the metal samples is not necessary, due to the fact that, in previous test series' carried out in accordance with TL 8135-0002 of the Bundeswehr², no significant differences were determined between samples which had been sanded, sanded and rinsed with demineralised water or sanded, rinsed and cleaned with methanol. During the preparation of the samples, care must be taken that they do not come into contact with palm sweat or with other impurities. The preparation of the metal samples should take place directly before the implementation of the test, in order to ensure that they have not already suffered from corrosion as a result of an exaggerated previous period of storage.

The metal samples are connected conductively with each other with the aid of a copper rail. The copper rail consists of a five-legged profile, thus meaning that the five metal sheets can be screwed to the rail to form the shape of a star. This guarantees a compact structuring of the test. Due to the fact that the required profile is not in a common trade format, it was constructed using four sheets of angled copper, screwed together and measuring 100 x 40 x 0.5 mm in size. The screw connections consist of synthetic bolts. Five centered holes are drilled into all the legs of the copper rail, close to the edges. At the points of contact with the metal samples, the copper rail is freed of oxide using 320er sandpaper, thus ensuring that a good conducting contact to the metal samples is created. The metal samples are then attached to the copper rail with the aid of synthetic nuts and bolts. The order of the metal samples in the envelope can be seen in diagram 2

A made-to-measure VCI strip is placed around each of the metal samples, as close as possible to the copper rail and fixed with a piece of adhesive tape (diagram 1).

² The TL 8135-0002, Edition 7 of the Bundeswehr essentially corresponds with the American Federal Standard 101C, Method 4031. The deviations are purely a result of the adjustment of the German measurements and standards

The same is carried out with PE-foil on the control the control sample. These strips enable the examination of the VCI contact effects.

Next, the foils are fastened to form an envelope. For this purpose, the short sides of the foil are placed over each other and welded together. The hose created in this way is then welded together at one open end, meaning that a sack with a volume of approximately 141 is created. The aid auxiliary apparatus, made of synthetic material, is then placed in the sack, on which the metal examination subjects of austenitic stainless steel which have been screwed together is resting, facing upwards³. Finally, the deionised water required for the condensation is fed in. Various variations for this were tried out in preliminary examinations. On the one hand, the water quantities were varied and, on the other hand, the means of feeding in the water. The experiments ranged from small dishes, paper soaked in water to pure water. The quantities of water must be sufficient to saturate the air, even at these burdening temperatures. The greater the surface area of water, the quicker the supply of water into the air during the phase of heating, thus meaning that the possibility of a short-term drop in the relative humidity in the test atmosphere is minimized. It became apparent that the optimum combination for this test consists of a paper towel, measuring 24 x 24 cm, folded twice and soaked in 10 ml of deionised water. Based on practical experience, this paper towel is comparable with hygroscopic packaging material containing moisture (i.e. wood). This paper towel is pushed underneath the auxiliary apparatus and the envelope is welded air-tight. In this context, care must be taken that the smallest possible quantity of air remains in the envelope, in order to ensure that, when the examination structure is subsequently heated, a bursting of the envelope as a result of expanding air is avoided. All of the described preparations are to be carried out as quickly as possible, so as to ensure that as little as possible of the VCI active principle escapes from the foil.

TEST PROCEDURE

VCI develop their protective effects on the surface of the metal. Therefore, they must first reach the metal surface over a VCI-saturated atmosphere. Due to the fact that the

VCI have a distinct steam pressure (10^{-2} - 10^{-4} mbar) and are generally comprised of various active principle components, the saturation of the atmosphere takes place through gradual sublimation. As this takes place over a longer period of time, the VCI is not yet capable of developing its protective effects immediately after the construction of the packaging. Resulting from the fact that the steam pressure varies from VCI to VCI, the

³ It became apparent in preliminary tests that the metal sample which is facing upwards is subjected to the lowest corrosive action - probably as a result of the condensed water which is running off. Due to the fact that the sample made of stainless steel never corroded during the preliminary tests, it is here subjected to the lowest corrosive action facing upwards

period of time which must be adhered to between the packaging and the first corrosive action, called the conditioning period, is also different from VCI to VCI. Due to the fact

that the steam pressure of the VCI is a function which is dependent on the temperature, the conditioning phase is also temperature-dependent. The VCI carrier material also has a further influence. The VCI foil often used mostly contains, on the one hand, less active principle as a VCI paper and, on the other hand, surrenders the contained VCI molecules to the air much slower. Therefore, in the use of VCI foil, a longer conditioning period should be calculated as for VCI paper.

As is apparent in the test description up until now, the conditioning phase takes place in saturated air - i.e. moist, due to the fact that the water has already been added to the atmosphere. Nonetheless, this neither has an influence on the protective effects nor on the duration of the conditioning phase of the VCI foil, as has already been determined within the framework of preliminary examinations⁴. The duration was set as being 20 hours.

The air of the test atmosphere is already saturated with water after a relatively short period of time. This can lead to premature condensation of the metal samples in cases of slight temperature fluctuations. This is even a desired effect in this instance. Due to the fact that, resulting from the large surface area in a proportionate comparison to the metal mass, large temperature differences between the surrounding air and the metal cannot be maintained, a premature condensation would only be very weak. However, even after a slight condensation, the generally water-soluble VCI located in the air is capable of satiating the condensed water. If the light condensed water film evaporates, more VCI molecules remain on the metal surface as previously was the case [2]. On the event of a second, more pronounced condensation, the protective effects of the VCI are therefore even greater than they would have been without the first weak condensation.

In the course of the further test implementation, care was taken that this advantage described with regards to the VCI protective effects took place. The first thermal burdening should only generate a very weak condensation on the metal samples. For these purposes, various experiments were carried out with regards to the required temperatures. On the basis of these, it was determined that the test apparatus should be stored, after a period of conditioning lasting 20 hours, for 30 minutes in an oven at 80°C. Despite the high temperature, only a slight condensation takes place, as a result of the fact that the water from the paper towel cannot be supplied with sufficient speed in order to saturate the test atmosphere over the entire period. There is therefore not even corrosion

⁴.During the preliminary examination, experiments were carried out with various phases of conditioning - moist (90% relative humidity) and dry (50% relative humidity). This was intended to provide conclusions regarding the evaporation kinetics of the VCI in dependence on the humidity. Due to the fact that foil is unable to soak up water, no influence on the build up of the saturated atmosphere could be expected. In cases of VCI papers, the water absorption of the paper, linked to the high humidity, has a negative influence on the evaporation speed

to be seen on the control sample. On removing the test structure from the oven however, the water condenses as a result of the drop in temperature on the interior surfaces of the entire foil. After 90 minutes of storage at room temperature, small drops of water are still clinging to the interior surfaces of the foil. If the test structure is then placed back into the oven at 80°C for 30 minutes⁵, the test atmosphere remains saturated,

even after the increase in temperature, as a result of the water supplied from the surfaces of the foil, i.e. a considerably stronger and also visible condensation, which corrodes the control sample.

TEST EVALUATION

The test evaluation takes place in several stages through assessment of the metal samples by the naked eye. Corrosion manifestations should be expected on the control sample on the St 37 and the galvanized steel sheet under the described test conditions. In this context, a slight discoloration of the brass and aluminum samples was not regarded as being corrosion damage. Both sides of the metal samples are examined for manifestations of corrosion, due to the fact that different results had been determined in this context during the preliminary examinations, which, however, did not appear to be subject to any regularity. The classification of the protective effects of the VCI takes place relative to the control sample and is allocated to one of the following categories for each type of metal:

- No corrosion-promoting effects
- Strong / slight corrosion-promoting effects
- No protective effects
- Slight / average / good protective effects

The classification under 'no corrosion-promoting effects' is selected if neither the control sample nor the VCI sample suffer from corrosion, due to the fact that, in such an instance, no statement can be made regarding the possibility of the existence of protective effects of the VCI. The possibility for classification is a strong, i.e. slight corrosion-promoting effect is necessary, due to the fact that VCI, in combination with certain metals, can also have a catalytic effect on the corrosion processes. Generally, no corrosion manifestations can be expected in the area of the contacts with the metal samples - also not on the control sample, due to the fact that no condensation occurred on these areas underneath the foil. Therefore, it is only necessary to note any observations which deviate this in the evaluation.

⁵ The duration of action of 30 minutes is completely sufficient. This was determined during preliminary tests, during which, after periods of action lasting in excess of 30 minutes, no significant increase in the corrosion manifestations was to be observed after longer periods of condensation.

RESULTS AND DISCUSSION

The test was carried out with two VCI foils, described hereafter as VCI A and VCI B. The difference between the two products consists in the manufacturing process. The same

VCI active principle was used, however foil A is a one-layer foil (mono foil) which let the VCI sublime on both sides of the foil. The foil B is a double layered foil (coex foil), in which the VCI active principle is only attached to one layer of synthetic material. The VCI therefore primarily sublimates in one direction, thus meaning that the direction of the relevant side of the foil towards the sample material must be adhered to.

Initially, the two products were examined with the aid of the TL 8135-0002 (7) in order to preserve the first impression of their protective effects. In this context, the foil A was classified as having 'average corrosion-protective effects' and the foil B was classified as having 'no corrosion-protective' effects. Variations with regards to the conditioning duration did not result in any improvement of the protective effects.

The examination which was subsequently carried out with the new test procedure achieved considerably better results (compare table 1).

The test results clearly demonstrate that both foils have a good to very good protective effect, despite the fact that this could not be expected on the basis of the tests which had been carried out previously.

In a more exact comparison of the test results, it becomes apparent that the TL 8135-0002 (7), attested in the evaluation the mono foil better protective effects than the coex foil. However, in theoretical practice at least, the exact opposite should be expected if the foil is used as the exterior packaging envelope. Because, as a result of the several layers of the foil, the outermost foil layer represents an additional blocking layer, which stops the VCI from escaping out of the packaging. This theory can only be confirmed by the presented test procedure, due to the fact that practical experience has been taken into consideration to a greater extent and the foil acts as a packaging envelope. Erroneously, the TL 8135-0002 (7) provided the opposite result, due to the fact that the test conditions favor the foil which emits the greatest numbers of VCI molecules - regardless of in which direction. It has no effects on the assessment of the test that the VCI molecules, in practice are actually escaping in the 'wrong' direction.

The good opportunities for assessment of the VCI protective effects by the test procedure presented is primarily a result of the choice of the test parameters. Great value was placed on aligning the parameters to the way the VCI works. Particular attention was paid to ensure that the concentration of VCI in the condensed water on the metal examination subject did not, at any time, drop below a critical value, beneath which a protective effect can no longer be maintained. Additionally, the condensation intensity of the metal examination subject was selected to be lower than in other test procedures, due to the fact that the VCI protective effects are also dependent on the electrolyte film thickness on the metal surfaces [2]. In this way, through the first weak condensation of the metal sample, the concentration of the VCI on the metal surface is increased. The protective effects are therefore greater during the subsequent, stronger condensations they would have been had there been no previous weak condensation and there had been an immediate strong condensation, as is the case in the majority of the known test procedures.

During the determination of the other test parameters, the requirements of practical experience were given particular consideration. In this context, the combination of various metals should be mentioned at the start. Additionally, i.e. by adding moist paper towels, the hygroscopic packaging material, partially contained in a package, is simulated. As is the case for most practical uses, the exterior packaging envelope does not consist of a glass container, rather of the VCI foil itself. The relationship between the VCI foil surface and the packaging volume has been sized generously, due to the fact that, in adhering to the so-called 30 cm gap rule, an oversizing of the VCI also takes place in practice. Due to the fact that it is generally impossible to exclude contact between the VCI foil and the metal surface which is to be protected, the test examined the contact effects of the VCI, in addition to the protective effects over the steam phase. The multi-layered test evaluation makes it possible to select in a targeted manner suitable VCI for different burdens, such as storage or dispatch overseas.

The test structure guarantees that, by employing slight modifications, the interests of the user can be taken into consideration: by leaving out or adding metal sheets, he can adjust the test to suit his own concrete case of use.

Despite the demands placed by the VCI and practical experience, the testing procedure is relatively simple and can be implemented quickly, due to the fact that the efforts required for preparation were kept to a minimum and the test results are available within 24 hours.

In addition to this string of advantages, there are however several disadvantages linked to the testing procedure presented. For example, the test is exclusively designed to examine VCI foils. Nonetheless, an alteration to suit other forms in which VCI is supplied should be possible without great efforts. Although, with regards to many interests, the testing procedure takes many requirements of the practice into consideration to a greater extent than previous tests, greater demands placed in the protective effects may arise in practice than those placed by the testing procedure in the VCI. In this context, an increase in the number of cycles can lead to a higher corrosive action. This is more effective than a prolonging of the condensation duration [3], which would also increase the action. The thermal burdening should not be increased under any circumstances, due to the fact that the risk exists that the VCI may otherwise decompose.

A further weakness is that the test results can only be transferred to goods which have the same metal combinations in similar quantities and with similar surface area proportions. Should this not be adhered to, then otherwise deviating proportions may lead to completely different results which would then also have consequences on the protective effects of the VCI. Naturally, further research must be carried out with regards to the purely coincidental corrosion manifestations.

The examination of the VCI contact protection effects does not take place in the determined corrosive action.

The VCI are not subjected to any aging during the test. Despite the fact that a gas escape of the VCI molecules through the foil occurs during the test implementation, the period is far too short for this to have a tangible effect. Therefore, should the protective effects of aged VCI be the subject of a test, then the aging must take place beforehand - either through synthetic or natural aging.

Principally, the testing procedure still has certain loopholes in the form presented, also with regards to the reproducibility, due to the fact that certain test parameters and test equipment have not been defined exactly. However, the target was not to present a normal testing procedure, rather to simply demonstrate new test approaches in order to do justice to the new VCI recipes and, at the same time, to provide the marketing with an opportunity of presenting the effectiveness of this method after its further development.

Should high transport, traffic volume and storage burdening occur, i.e. should large quantities of condensed water be generated during the first condensation, then this method will fail. In these instances, tests must be carried out in a changing climate on real components, in order to preserve process security. This is proved by damage which occurred in recent times in the increased component tourism.

CONCLUSIONS

The test procedure presented in this paper is capable of validating the protective effects of VCI foils in connection with various, electrically conductive, connected metals.

As a result of test conditions which were specially designed to match the way in which VCI works and the combination of various metals, this testing procedure is capable to a much greater extent of putting the protective effects of VCI to the test than the test which is generally recognized in Germany, the TL 8135-0002 (7).

In addition to the objective of aligning the test parameters to a validation of VCI protective effects, many aspects were incorporated into the testing procedure which have strong relevance to the practice. These are not included in the other testing procedures which currently exist.

Nonetheless, the results of the test result should not be consulted as the sole criterion for the selection and assessment of VCI, due to the fact that, under practical conditions, the corrosive action may be considerably greater than that generated in the test. In practice, different sizes, weights and forms of the samples used in the test and the actual packaging item influence the VCI protective effects, thus meaning that further tests would certainly be recommendable, i.e. in the form of field or laboratory examinations on finished components.

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	test assembly with VCI		VCI A	VCI B
	top	bottom		
stainless steel	free of corrosion	free of corrosion	no corrosion stimulating effects	no corrosion stimulating effects
galvanized steel	few corrosion products	several corrosion products	very good corrosion inhibiting abilities	good corrosion inhibiting abilities
brass	stain	stain	no corrosion inhibiting abilities	no corrosion inhibiting abilities
aluminum	stain	stain	no corrosion inhibiting abilities	no corrosion inhibiting abilities
steel	several corrosion products	several corrosion products	very good corrosion inhibiting abilities	good corrosion inhibiting abilities

Table 1

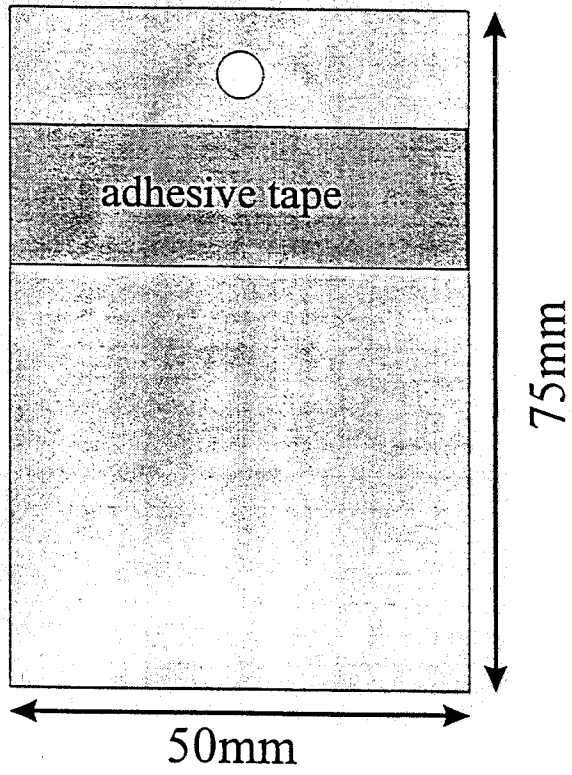


Figure 1

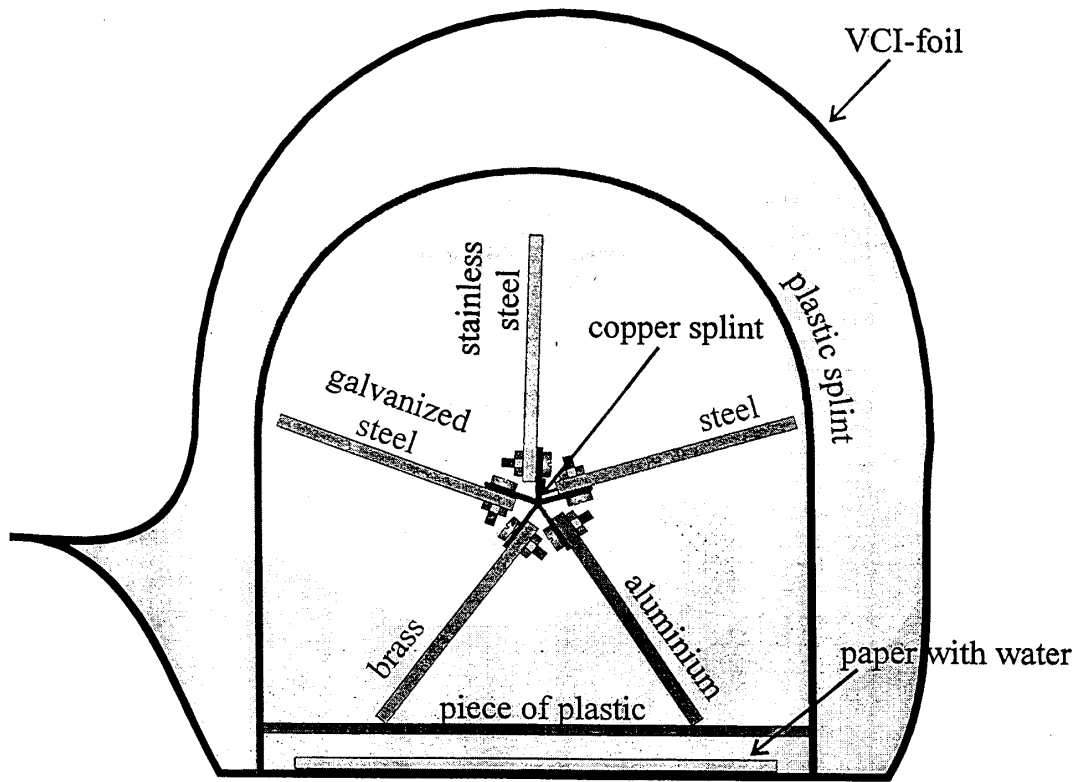


Figure 2