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AUTHOR

C. A. Burgess

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This document consists
of 10 pages.

A STANDARD ALS1 BRAZE WETTING TEST

FOR ALUMINUM COMPONENTS USED IN THE LEAD-DIP PROCESS

by

C. A. Burgess

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FUELS PREPARATION DEPARTMENT

By Authority of PR-24

WASnyder, 2-23-94. February 1, 1961

By Gerri Maley 31094

Verified By J.E. Savelly 3-15-94

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A STANDARD AlSi BRAZE WETTING TEST

FOR ALUMINUM COMPONENTS USED IN THE LEAD-DIP PROCESS

INTRODUCTION

The wetting of the aluminum components by the aluminum-silicon brazing alloy is a major factor influencing the resulting quality of the fuel elements. Several process variables associated with the impact extrusion of aluminum components influence the resulting surface characteristics of the aluminum. In addition, these processing variables interact with the HAPO component production cleaning process to give varying results. The purpose of this document is: (1) to define the parameters for selecting, preparing, testing and evaluating the AlSi wetting potential of aluminum components used in the lead-dip process; and (2) to provide a tool for measuring the acceptability of aluminum components for the preparation of fuel elements.

Aluminum can-component lots must have an average AlSi braze wetting potential of Rank 7 or greater to be acceptable.

DISCUSSION

In order that the as-received quality of extruded aluminum components can be defined relative to the AlSi brazing process, a braze-wetting test is proposed. This test will enable the Fuels Preparation Department to screen all as-received aluminum component shipments for their braze-wetting potential. The following may develop from such a program:

- a. Establish quality levels of the various vendors.
- b. Aid in developing new vendors and determining when their process is acceptable.
- c. Explore the use of new extrusion lubricants.
- d. Explore the effect on HAPO fuel element quality of processing variations at the vendor's plant.

Lot Sample Selection

A lot is defined as all of those components received at HAPO on a given calendar day of a specific model type from a given vendor. (The component will be referred to as a can throughout the balance of this report.) In general, this means a lot will usually consist of from 1000 to 100,000 cans. The quality of a given lot in all of the following discussion is intended only to mean that characteristic of AlSi wettability.

Based on past experience, a sample of 20 cans will normally give the required protection against accepting a poor quality lot, while at the same time insuring that acceptable lots have a high probability of being accepted. This assumes

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that the cans in a given lot are homogeneous. However, due to a lack of population homogeneity, this sample size must take into consideration population stratification. Also, the sample size must be controlled to afford the greatest economy and be of a convenient size. Past experience with this test indicates that a maximum sample of 100 cans should apply, with the minimum being 20 cans.

A suggested sampling plan would be as follows:

<u>Component Lot Population</u>	<u>Sample Size</u>
<5000	20
5000-10,000	40
10,000-100,000	80
>100,000	100

The sample should be randomly selected from a maximum number of boxes throughout the lot. This sample will then be subjected to the AlSi wettability test. Each component will be graded and numerically ranked according to the non-wet scale defined later in this report. If a decision to accept or reject the lot is not reached, a second sample should be taken.

A decision can normally be reached with the first sample and it is extremely unlikely that more than two samples will be required, but the plan provides for taking as many sequential samples as deemed necessary.

It has been determined that component lots having an average wettability rank (μ) of less than seven are not acceptable for the existing lead-dip process. It has also been determined that component lots having an average rank (μ) of seven or greater wet well with AlSi during fuel element assembly.

In detail, the plan proposed is to obtain the average rank for the sample, e.g. 20 components. If this average exceeds the upper value given in Table I below, accept the lot. If this average is less than the lower limit, then reject the lot. If it is between 7.30 and 8.02, sample an additional 20, compute the over-all average of the 40, and be guided by the acceptance criteria in Line 2 of the table. This procedure is repeated until a decision is reached.

TABLE I

ACCEPTANCE CRITERIA FOR SEQUENTIAL SAMPLING PLAN

<u>Sample Size</u>	<u>L.L.</u>	<u>U.L.</u>
20	7.30	8.02
40	7.52	7.88
60	7.60	7.84
80	7.63	7.82
20 n	$7.75 - \frac{0.45}{n}$	$7.75 + \frac{0.28}{n}$

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This particular plan has the following properties:

- I. Letting μ equal true rank average for the lot, the following relationship exists between μ and the probability of rejecting the lot:

<u>True Average Value</u>	<u>Probability of Rejecting Lot</u>
$\mu_0 = 8.5$	0.01
$\mu_1 = 7.0$	0.95

Note that this gives a very low probability of rejecting a lot with an average rank as large as 8.5, or of accepting a lot with an average rank less than 7.0.

II. Average Sample Number

Letting a sample of 20 cans consist of one experimental unit, we find the average sample numbers required as a function of μ :

<u>μ</u>	<u>ASN</u>
8.5	< 20 Components
7.75	20 < ASN < 40 Components
7.0	< 20 Components

This implies that unless the quality is borderline, the initial sample size of 20 cans should be adequate to reach a decision. For borderline material two groups of 20 cans each will normally be all that is required.

III. Sources of Variation

A study was completed within the scope of this proposed wetting test. A total of 1000 samples were selected from ten different can lots. These can lots were selected from various models and were representative of the can inventory during December 1960.

a. Measurement Error

The statistical test indicated that there is no significant difference between inspectors. This allows an estimation of the measurement error. These estimates are:

$$\sigma_e^2 = 0.80$$

$$\sigma_e = 0.90$$

per individual can. For an average of 20 component wettability measurements the error due to measurement is reduced to:

$$\sigma_e = \frac{0.9}{\sqrt{20}} = .20.$$

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The measurement error increases as the quality decreases.

b. Lot-to-Lot Variation

If the quantities $\alpha_1, \alpha_2, \alpha_m$, are the true wettability averages in different lots from the same vendor, then the variation in these averages is equal to:

$$\sigma_{\alpha}^2 = 0.047$$

$$\sigma_{\alpha} = 0.22.$$

c. Within-Lot Variation

A wettability measurement X, from one lot, is made up of two components as follows:

$$X = \beta + e$$

where β is the true wettability value and e is the measurement error. (With unbiased measurements average e = 0.)

Then

$$\sigma_X^2 = \sigma_{\beta}^2 + \sigma_e^2$$

The variance of β is estimated to be

$$\hat{\sigma}_{\beta}^2 = 2.1$$

so that the variance of x sampled from one lot is estimated to be

$$\hat{\sigma}_X^2 = 2.1 + 0.8 = 2.9$$

$$\hat{\sigma}_X = 1.7.$$

d. Variation in Average Values

The variance in individual values of X from one vendor is estimated to be

$$\begin{aligned}\hat{\sigma}_X^2 &= \hat{\sigma}_{\alpha}^2 + \hat{\sigma}_{\beta}^2 + \hat{\sigma}_e^2 \\ &= .0047 + 2.1 + 0.8\end{aligned}$$

Note that the largest contribution to the variance is due to the variation between components within lots. For $\bar{X}$, obtained for m lots with n components per lot, and S measurements per component, the variance is then

$$\hat{\sigma}_{\bar{X}}^2 = \frac{\hat{\sigma}_{\alpha}^2}{m} + \frac{\hat{\sigma}_{\beta}^2}{mn} + \frac{\hat{\sigma}_e^2}{mnS}$$

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$$= \frac{0.047}{m} + \frac{2.1}{mn} + \frac{0.8}{mnS}$$

Thus for the variance of the average of a sample taken from 10 lots, with 100 samples per lot and 2 measurements per sample, the variance is

$$\frac{\sigma^2}{\bar{X}} = 0.0047 + 0.0021 + 0.0004$$

$$\frac{\sigma^2}{\bar{X}} = 0.0072$$

and $\frac{\sigma}{\bar{X}} = 0.085$

Component Preparation

The selected sample will be randomly mixed and the bottom of the component coded with an appropriate designation (e.g., lot number). The components will then be divided into at least three, but not more than five, sub-groups. These sub-groups are then coded numerically by number and run through the cleaning line in sequence to minimize the effect of cleaning line operational variables. There shall be a time limit of 50 seconds maximum between each stage of the cleaning operation. The cleaning shall consist of the following sequence of operations:

I. Component Degreasing

a. Agent, trichloroethylene.

b. Equipment

1. Hot stage boiling submerge or hot spray, Temp. 86-95 C, Time 50 seconds minimum.
2. Cold Stage Cold Submerge or Cold Spray, temp. 35 C maximum, Time 50 second minimum.
3. Vapor stage - hot vapor condensing on the surface and draining freely 86 to 95 C, Time - 50 seconds minimum.

c. Objective: Remove oils and waxes by dissolution.

II. Cold Water Spray

a. Agent, cold process water, Temp. 30 C maximum, time 50 seconds.

b. Equipment, spray tank or tank submerge.

c. Objective: Lower the temperature of the components in preparation for alkaline cleaning.

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III. Alkaline Cleaning

- a. Agent, Wyandotte, Altrex.
- b. Equipment, soaking tank, temp. 75 ± 5 C, concentration 4-5 oz./gal., time 12 ± 3 minutes.
- c. Objective: Clean by detergent action.

IV. Hot Water Rinse

- a. Agent, hot process water
- b. Equipment, tank and spray, temp. $60 \text{ C} \pm 5 \text{ C}$.
- c. Objective: Remove alkaline cleaning agent from the surface of the component.

V. Cold Water Rinse

- a. Agent, cold process water, temp., 30 C maximum.
- b. Objective: Remove alkaline material from the surface of the component and to lower the surface temperature of the component before it enters the deoxidizing stage. It has been shown that high temperature deoxidation is not desirable for this surface preparation.

VI. Deoxidation

- a. Agent, Diversey 514
- b. Equipment, soaking tank.
 - 1. Temp. $20 \text{ C} \pm 10 \text{ C}$.
 - 2. Concentration 32 ± 3 oz./gal.
 - 3. Etchant activation exposure X-8001 alloy 1 sq. ft./gal. of solution for 15 minutes.
 - 4. Etchant life exposure to ^{32.0}~~200~~ sq. ft./gal. X-8001 alloy for 15 minutes.
 - 5. Time 7 to 15 minutes.
 - 6. pH controlled between .3 and .1 on the standard scale.
- c. Objective: Removal of the existing aluminum oxide surface and its replacement by a unique oxide that lends itself to good brazing properties.

VII. Cold Water Rinse

- a. Agent, cold process water.

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- b. Equipment, soak tank, temp. 30 C maximum, time 50 seconds minimum.
- c. Objective: To remove excess Diversey solution. The temperature should be held to minimum, otherwise the unique oxide film being formed will be undesirably altered.

VIII. Cold Water Rinse

- a. Agent, cold process water.
- b. Equipment, soak tank, temp. 30 C maximum, time 50 seconds minimum.
- c. Objective: Remove any remaining Diversey solution. The unique oxide film has formed by this stage of the process and the temperature is not quite as critical.

IX. Methanol Rinse and Drying Agent

- a. Agent, commercial grade methanol.
- b. Equipment, soak tank, a maximum of 18% H₂O, maximum sp. gr. .842 at 18 C.
- c. Objective: Additional rinsing and a drying medium.

X. Drying

- a. Agent, forced warm air circulation.
- b. Equipment, conventional forced warm air drier, temp. 70 C maximum, drying time 30 minutes maximum.
- c. Objective: Assurance that no residual water or other volatile substance will be present on the surface of the components during brazing.

The components are removed from the drier as a series of marked runs. These runs are randomly mixed as they are placed in steel canning sleeves. The component-sleeve assemblies are then sent to the brazing furnace for performance of the AlSi braze wetting test. A maximum time limit of four hours shall be adhered to between drying and performance of the braze wetting test.

Brazing Furnace Operating Characteristics

I. Brazing Medium

- a. Aluminum-silicon alloy of the following chemical composition:
 - 1. Silicon 11.3% + 0.5%
- 0.8%.
 - 2. Nickel 0.5% maximum.

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3. Aluminum plus normal impurities as determined by the Fuels Preparation Department Analytical Laboratory Analysis.

b. Temperature: The brazing bath temperature will be 1 to 2 C above the first thermal arrest point of the bath metal. (Note: The first thermal arrest point is defined as the eutectic temperature (578 C) plus the temperature spread as determined by thermal analysis for the silicon concentration of the bath metal.)

c. Brazing Cycle:

1. The sleeve-can assembly will be placed in a brazing basket-holder such that the assembly extends not more than 5/8 of an inch above the bath surface. The can-sleeve assembly will be placed in this preheating position for a period of 47 ± 3 seconds.

2. The can-sleeve assembly is then submerged below the surface of the bath and the can component is filled with molten bath metal. The can-sleeve assembly is held in the submerged position for a period of 36 ± 3 seconds. The brazing basket holder has a clamping device that clamps the can-sleeve assembly. The entire assembly is vibrated in the submerged position to aid in gas removal and to improve component wetting. The vibration starts five seconds after submerge and is continuous for a period of 29 ± 4 seconds.

The vibration is induced into the system by a Peterson Vibrolator Model BD-13.* The frequency shall be maintained between 12,000 and 20,000 vibrations per minute in the vertical plane.

At the completion of the submerge cycle, the can-sleeve assembly is removed from the bath and the excess molten metal is poured from the can. The can-sleeve is then quenched in water in such a manner that water does not enter the can and the can-sleeve is quenched externally.

The time from the end of the submerge cycle to quenching shall not exceed 15 seconds.

The can component is then removed from the steel sleeve and prepared for evaluation.

Sample Evaluation

The cans are then sawed in half on a band saw equipped with an appropriate aluminum cutting blade. The can is folded open in order to fully expose the brazed surface. The curved can halves are then flattened by the use of a rubber or plastic mallet to loosen any non-adhering braze metal, and to facilitate the inspection of the surface. A plastic grid divided into one-half centimeter squares is laid over the

* A product of the Martin Engineering Company, Neponset, Illinois.

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brazed surface and the total non-wet area is graphically integrated for both halves of the individual can components. The can may be flexed if an area is suspected of being non-wet. A ranking number is then assigned to the total number of non-wet areas as follows:

<u>Rank</u>	<u>Number of Sq. Non-Wet</u>	<u>Area of Non-wetting</u>
10	0	0
9	0-1	.25 cm sq
8	1-4	1.00 cm sq
7	4-9	2.25 cm sq
6	9-16	4.00 cm sq
5	16-25	6.25 cm sq
4	25-36	9.00 cm sq
3	36-64	16.00 cm sq
2	64-100	25.00 cm sq
1	100-144	36.00 cm sq
0	144-up	gross non-wet

After this ranking number has been determined, the code on the bottom of the can component is noted and both the ranking and code number are recorded. This eliminates any biasing in ranking that might occur in tests where it is necessary to run both a control group and a test group for comparison studies. Through experience, it has been observed that there is a high degree of replication between inspectors and therefore it is usually only necessary to have one inspector and one recorder evaluate the samples. In some instances where the dollar involvement is high and rejection is probable, it would give additional strength to the statistical evaluation if two inspectors are used.

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C. B. Jaech
Engineer
ALSI Product Engineering

DATE

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8/9/94

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