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TITLE

Hydrofluosilicic Acid as a Cap and Can Etchant

AUTHOR

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E. W. O'Rorke
Superintendent, Process Unit
METAL PREPARATION SECTION

March 17, 1953

HYDROFLUOSILICIC ACID AS A CAP AND CAN ETCHANT

Introduction

Aluminum caps and cans are thoroughly cleaned, before being used to can slugs, to insure wetting of the metal surfaces by molten AlSi in the canning pot. An acid bath is used, as part of the cleaning operation, to remove surface oxide and other surface films from the metal. Two acid solutions are authorized in the standard operating procedure: a 20% phosphoric acid solution for etching both caps and cans, and a 1% hydrofluosilicic acid solution to be used for caps only. It is desired to determine the feasibility of using hydrofluosilicic acid exclusively as an etchant for both caps and cans.

Summary

Wettability measurements (i.e., measurements of amount of surface film) were made on caps cleaned in 20% phosphoric acid and in hydrofluosilicic acid solutions of up to 4% concentration. The hydrofluosilicic acid etching gave better wettability than did phosphoric etching. Process tests were run in which slugs were canned using caps and cans etched in 1% and 3% hydrofluosilicic acid. Satisfactory wetting and canning results were obtained, which compared favorably with results from the use of phosphoric acid.

It was found that a 1.3% hydrofluosilicic acid solution is the optimum concentration if the present 100-gallon stainless steel acid tank is used. This 1.3% optimum is based on a recommended procedure of mixing a fresh batch of solution every morning, and using each batch for a 16-hour period.

The cost of commercial grade hydrofluosilicic acid, if used in the manner suggested, would be about $23.00 per 6-day week; as compared to about $46.00 per week for phosphoric acid as it is presently being used. These cost figures are based on the f.o.b. factory prices.

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Recommendations

The following recommendations are based on results of the investigation described in this report:

1. It is recommended that all cap and can etching be carried out in the acid tank now in use, using batches of hydrofluosilicic acid with initial concentrations of 1.3%.
2. Each batch of acid should be freshly mixed every morning and used for a 16-hour period. The used solution should be discarded at the end of every swing shift.
3. Etch times of three minutes or more are recommended. About ten minutes gives the most effective film removal.
4. Hydrofluosilicic acid is not recommended for etching caps only, since two acid tanks would be required, one for caps and one for cans.

Procedure, Results and Discussion

It is suspected that caps and cans etched in phosphoric acid retain an aluminum phosphate film on their surfaces. This phosphate film would reduce wetting of the metal in molten AlSi and might also cause welding difficulties. To test this, and to determine suitable acid concentrations and etch times, measurements were taken of the electrical surface resistances of caps etched in phosphoric and hydrofluosilicic acids. The resistance of the surface film is a measure of wettability, a low surface resistance indicating good wettability. This relationship was found to hold true on caps tested for wettability by placing them in the canning pot for the specified preheat time. Surface resistances were measured on caps etched for different periods of time. Hydrofluosilicic acid concentrations of 1, 2 and 4% were used. Results of these measurements are shown plotted in Graph No. 1. All of the caps etched in hydrofluosilicic acid showed better wetting characteristics than those etched in phosphoric acid.

Process tests were run in the cap and can room using 1% and 3% hydrofluosilicic acid solutions. Etch times of 8 minutes were used with the 1% acid (as per standard), and 2 minutes with the 3% acid. The solutions were placed in a polyethylene-lined tank. The following procedure was used: 8" caps were degreased and treated in normal manner by the cap and can operators, except for acid etch. The caps were etched in the prepared solutions by Quality Control personnel using batches of 20 or 30 caps each. Between batches, samples of solution were taken for analysis. After the samples were taken, a few cans were etched for canning. The etched caps and cans were rinsed and dried in normal manner.

The process tests were continued by having 225 eight-inch slugs canned with the following:

1. 190 slugs with Alcoa caps etched in 1% acid for eight minutes as per standard.
2. Ten slugs with both caps and cans (Alcoa) etched in 1% acid for eight minutes (not according to standard).
3. Ten slugs with Hunter-Douglas caps and cans etched in 1% acid for eight minutes.
4. Ten slugs with Alcoa caps and cans etched in 3% acid for two minutes.
5. Five slugs with Hunter-Douglas caps and cans etched in 3% acid for two minutes.

The 190 slugs with caps cleaned in 1% acid (Item 1 above) were canned according to standard; therefore, the good pieces were handled as normal slugs for charging into the pile. The rejects were transferred to Quality Control along with the 35 rejects (Items 2,3,4, and 5 above) that were not canned according to standard. All the reject pieces were stripped and examined for non-wet cap and can areas. The results were as follows:

Acid Etch	Caps	Cans	Type of Canning	Type of Cap or Can	Non-Wets
1%	130		triple dip	Alcoa	1
1%	70		lead dip	Alcoa	0
1%		10	lead dip	Alcoa	0
1%	10		lead dip	Hunter-Douglas	6
1%		10	lead dip	Hunter-Douglas	4
3%	10		triple dip	Alcoa	0
3%		10	triple dip	Alcoa	0
3%	5		triple dip	Hunter-Douglas	3
3%		5	triple dip	Hunter-Douglas	2

From these results it can be seen that the Alcoa caps and cans wet satisfactorily after either 1% or 3% etch with but one exception, while about half of the Hunter-Douglas caps and cans did not wet completely (Hunter-Douglas caps and cans also wet poorly with phosphoric acid).

The acid samples were submitted to the analytical laboratory for analysis. Laboratory results, volumes of solutions used for etching, and other recorded data were used to calculate the percentages of acid in solution as a function of the number of caps and cans that could be etched in the tank now in use in the cap and can room. This tank is of approximately 100 gallons capacity. The calculated figures are shown plotted in Graphs 2 and 3. The figures on the abscissas of the graphs represent caps and cans processed together in equal numbers.

Consultation with E.A. Smith, who had experience in using the 1% acid several years ago, revealed that use of this solution was discontinued because of the necessity of changing the solution too frequently. However, at that time, only 10 liter (or 2.64 gallon) batches were used. It was revealed also that the eight hour maximum standing time for the solution, as specified in the operating standard, was necessary because of deterioration or decomposition of the acid in water upon standing. It was found in the above tests, however, that allowing the solution to stand overnight, and using it the next morning, still gave good results in canning. This seems reasonable in view of the fact that the acid is marketed as a 30% solution in water.

Assuming, from the results of the tests, that a solution could be used for 16 hours (a fresh batch every morning), then a 1% solution in the present 100-gallon tank, used to etch 2700 caps and 2700 cans, would decrease in acid concentration to about 0.7% (based on results from analytical laboratory). Acid concentrations of from 0.9% to 3% give good results, as indicated by the tests and by the operating standard. If an initial concentration of 1.3% or more were used, then processing of 2700 caps and 2700 cans could be accomplished without the acid concentration becoming dangerously low. The cost of phosphoric acid as it is presently used is about $46.00 per six-day week, based on the at-factory price (18.4¢/lb. of 85% acid). This figure assumes that a fresh solution is made up once a week and the old solution discarded. The cost of using a 1.3% solution of commercial grade hydrofluosilicic acid, made up fresh daily, would be about $23.00 per six-day week based on the at-factory price (10.5¢/lb. of 30% acid).

Preliminary investigation has shown that either a titration method or use of a calibrated Wheatstone bridge (sol-u-bridge) should be a satisfactory means of checking acid concentrations during operation.

The only additional safety hazard would be in the handling of the concentrated acid (30%), which is dangerous if brought into contact with the skin.

Acknowledgment

The writer wishes to acknowledge the assistance of E.A. Weakley in the use of equipment belonging to Pile Fuels Technology. The equipment was used to obtain the surface resistance measurements described in this report.

D. S. Dixon

Process Unit
METAL PREPARATION SECTION

DS Dixon:es

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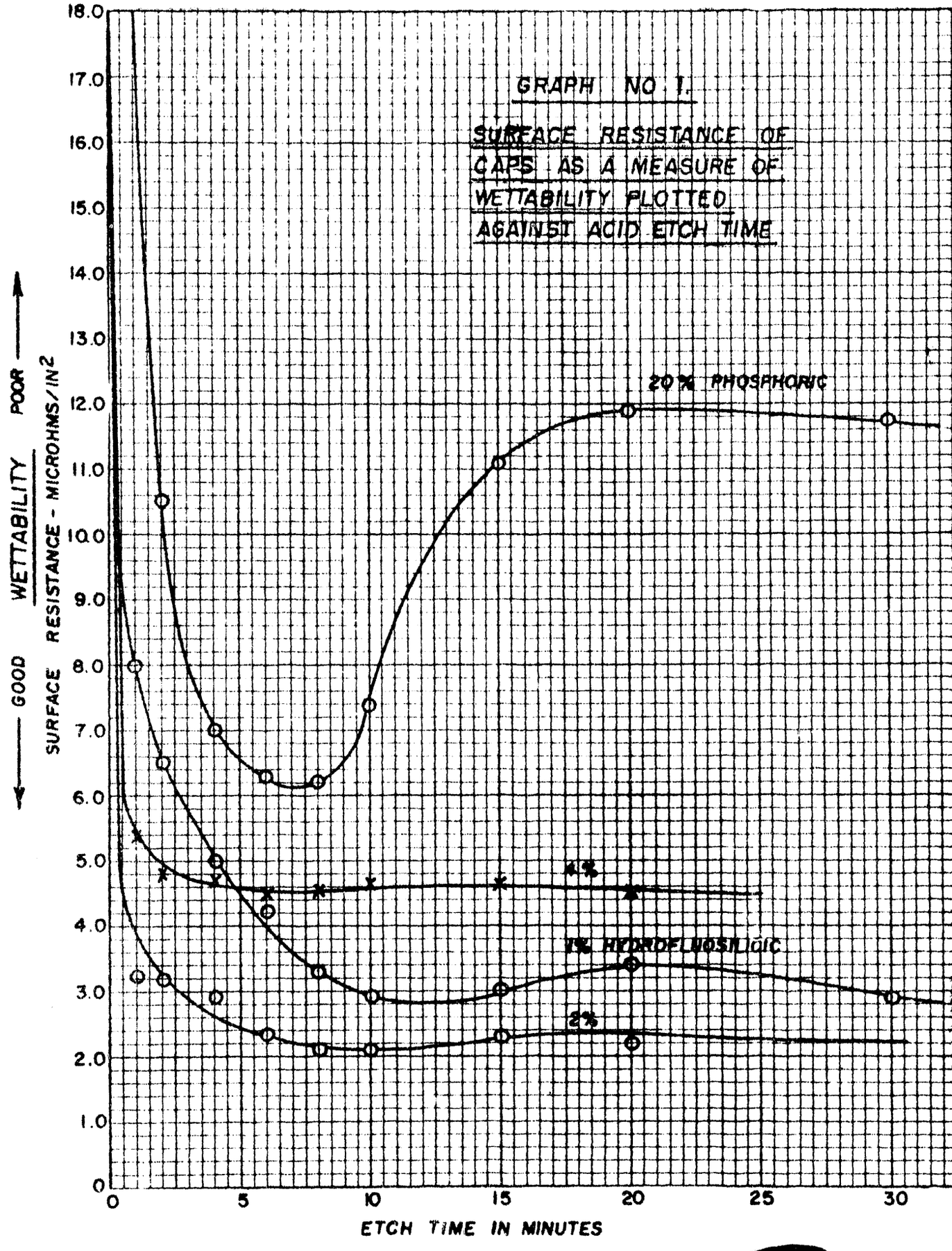
GRAPH NO 1.
SURFACE RESISTANCE OF CAPS AS A MEASURE OF WETTABILITY PLOTTED AGAINST ACID ETCH TIME
20% PHOSPHORIC
4%
1% HYDROFLUOSILICIC
2%
GOOD WETTABILITY POOR
SURFACE RESISTANCE - MICROHMS/IN2
ETCH TIME IN MINUTES
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
13.0
14.0
15.0
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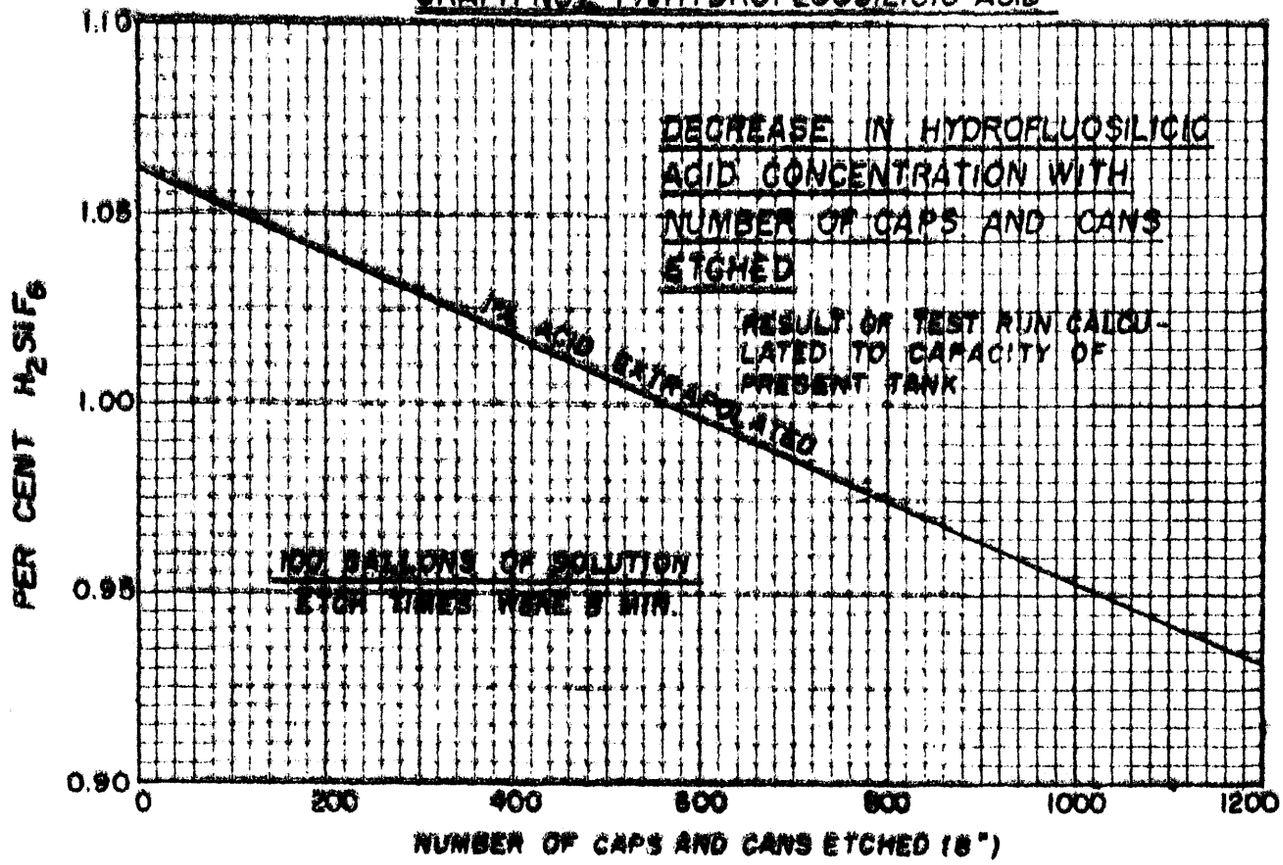
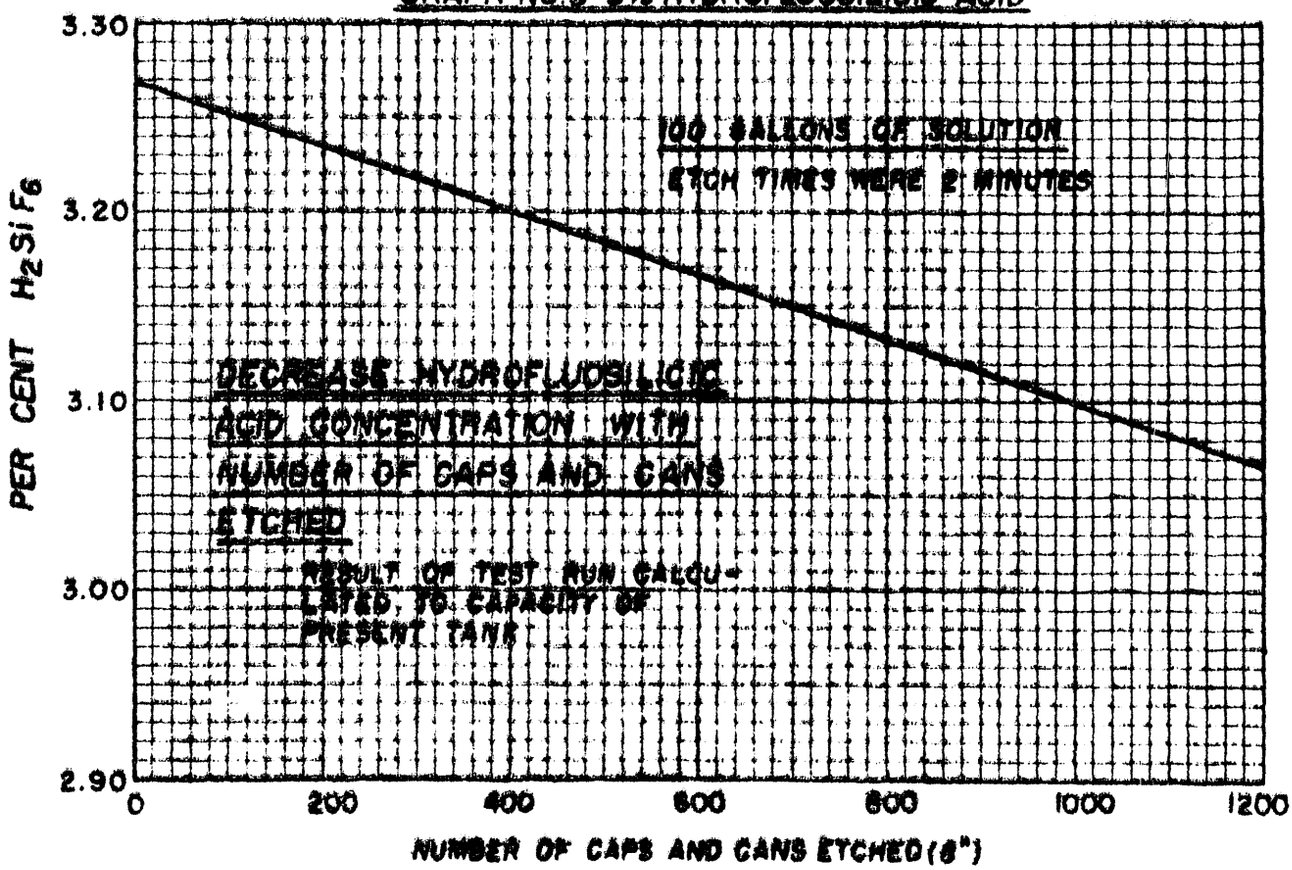
0
5
10
15
20
25
30

GRAPH NO 2 - 1% HYDROFLUOSILICIC ACID



GRAPH NO 3 - 3% HYDROFLUOSILICIC ACID



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