Technical Process Bulletin

Technical Process Bulletin No. 1011
This Revision: 06-09-2001

DEOXIDIZER 6/16

1. Introduction:

The DEOXIDIZER 6/16 process primarily utilizes two liquid products, Deoxidizer 6 Makeup and Deoxidizer 16 Replenisher. These are added to either dilute nitric acid or dilute sulfuric acid to produce an efficient production bath for the deoxidizing, desmutting or acid etching aluminum and its alloys.

The DEOXIDIZER 6/16 nitric acid bath is suitable for use in most applications, particularly where water rinsing is marginal, or on assemblies or fayed surfaces.

The DEOXIDIZER 6/16 sulfuric acid bath is especially suitable for deoxidizing aluminum for spot-welding. The surface resistance of the work so treated is low and remains low enough for several days to permit satisfactory spot-welding.

When a high percentage of high copper alloys are deoxidized or desmutted, buildup of dissolved copper in the bath, of as low as 200 ppm, can result in redeposition of metallic copper on parts particularly when titanium or stainless steel racks are used. At this point the DE7-17 Toner can be added to the bath to precipitate the dissolved copper.

DEOXIDIZER 6/16 baths impart a slight etch on aluminum alloys and bath conditions can be adjusted to bring the etch rate within customer specifications.

DEOXIDIZER 6/16 is an approved process for BAC 5765, Solution 27 (A,B & C).

2. Operating Summary:

General Use DEOXIDIZER 6/16 - Nitric or Sulfuric Acid Bath Makeup:

Chemical:	Bath Preparation per 100 Gallons:	
Acid	Nitric Acid 10 gallons	Sulfuric Acid 5 gallons
Deoxidizer 6 Makeup	5 gallons	5 gallons

BAC 5765 DEOXIDIZER 6/16 - Nitric or Sufuric Acid Bath Makeup:

Chemical:	Bath Preparation per 100 Gallons:		
BAC Soln. 27A	Nitric Acid 10 gallons	Sulfuric Acid 0 gallons	Deox 6 7 gallons
BAC Soln. 27B	0 gallons	5 gallons	7 gallons
BAC Soln. 27C	10 gallons	0 gallons	4 gallons

Operation and Control:

Deoxidizer Titration	14 to 24 ml
Nitric Acid Titration (10%)	3.0 ± 0.3 ml
Sulfuric Acid Titration (5%)	3.5 ± 0.3 ml
RT - DT Value, without DE 7-17 Toner	30 ml max.
RT - DT Value, using DE 7-17 Toner	70 ml max.
Etch Rate (2024 Clad alloy)	0.1- 0.4 mils/surf./hr.
Bath Temperature	60° to 90° Fahrenheit (15.6° to 32° Celcius)
Time	
Spray:	30 sec. to 3 minutes
Immersion:	1 to 10 minutes

3. The Process:

A complete process sequence normally consists of the following steps:

A. Cleaning
B. Water rinsing
C. Treating with the DEOXIDIZER 6/16 Working solution
D. Water rinsing
E. Further processing

4. Materials:

Deoxidizer 6 Makeup
Deoxidizer 16 Replenisher
Deoxidizer DE7-17 Toner
Additive A
Nitric Acid, 42° Bé
Sulfuric Acid, 66° Bé
Testing Reagents and Apparatus

5. Equipment:

The tanks for the DEOXIDIZER 6/16 bath may be constructed of 300 Series stainless steel (Type 316 preferred for weldability) or lined with high density polyethylene or similar acid-resistant materials.

6. Surface Preparation:

Most work can be cleaned using a suitable RIDOLINE® cleaner. If the work is heavily soiled and additional cleaning power is needed, a RIDOSOL® detergent cleaner additive may be added to the RIDOLINE cleaner bath. For situations where a degreaser is required, a product such as Ridoline 4355 can be used as an aqueous degreaser/precleaner.

The work, after cleaning, should be rinsed thoroughly with water. This rinse should be continuously overflowed to avoid contamination.

NOTE: Our representative will recommend the proper type of cleaner for each processing line.

7. Treating with the DEOXIDIZER 6/16-Working Solution:

Buildup:
Fill the tank three-fourths full with cold water. For each 100 gallons of final solution volume slowly add 10 gallons of 42° Bé nitric acid for the DEOXIDIZER 6/16 nitric acid bath **or** 5 gallons of 66° Bé sulfuric acid for the DEOXIDIZER 6/16 sulfuric acid bath and mix thoroughly. Add, while mixing, 5 gallons of Deoxidizer 6 Makeup. Add sufficient water to bring the solution up to the working level.

For use under BAC 5765, Solutions 27A or 27B, increase the makeup addition of Deoxider 6 to 7 gallons per 100 gallons of final solution volume. For use under BAC 5765, Solution 27C, reduce the makeup addition of Deoxider 6 to 4 gallons per 100 gallons of solution.

Operation:

Time/Spray:	30 seconds to 3 minutes
Time/Immersion:	1 to 10 minutes
Temperature:	60° to 90° Fahrenheit
	15.6° to 32° Celcius

The DEOXIDIZER 6/16 nitric acid or sulfuric acid bath works very effectively at ambient temperatures. Heating of the bath is not recommended.

The time required for deoxidizing depends on the amount and nature of the oxide. Natural oxide usually is removed in 1 to 3 minutes; heavier, heat-treat oxide may require 5 to 10 minutes. Deoxidizing prior to spot welding is usually accomplished in 3 to 5 minutes for most alloys. Immersion application usually requires more time than spray application. The bath should be mildly agitated in order to obtain greater surface uniformity.

8. Testing and Control:

Acid Titration:
Pipet two 2 ml samples of the DEOXIDIZER 6/16 acid bath into two beakers and dilute to approximately 100 ml with either distilled or deionized water. Add 10 drops of Indicator 2 to each sample. One of the two samples is used as a blank so that it is easier to distinguish the color change in the dark-colored solution.

Titrate one sample with Titrating Solution 89, while stirring the sample, until a blue-green color appears. Compare with the blank sample during the titration so that the development of the blue-green color will be more apparent.

The mls of Titrating Solution 89 used is the Acid Titration.

In very heavily loaded baths where the determination of the blue-green endpoint is extremely difficult, a potentiometric titration is recommended using a pH of 3.7 as the end point of the titration.

Acid titration range: 3.0 ± 0.3 mls for bath containing 10% nitric acid.
3.5 ± 0.3 mls for bath containing 5% sulfuric acid.

The acid concentration may be increased or reduced to achieve desired results. For concentrations other than those specified, maintain the Acid Titration range within ± 0.3 ml of that obtained on bath preparation.

To increase acid titration 1 ml: Add 3.4 gallons of 42° Bé nitric acid or 1.3 gallons of 66° Bé sulfuric acid per 100 gallons of bath volume.

Deoxidizer Titration:
Pipet a 5 ml sample into an iodimetric flask and dilute to approximately 100 mls with water. Add 1/2 teaspoon (approximately 1 g) of Reagent 2 and agitate to dissolve. Add about 15 ml of Reagent Solution 49. Stopper the flask and allow to sit for about one minute.

After the sample has set for approximately one minute, titrate with Titrating Solution 104 until a straw color is obtained. Do Not rezero the buret. Add several mls of Indicator 10, stir, and continue the titration until the blue-black color disappears for at least one minute. The mls of Titrating Solution 104 used is the Deoxidizer Titration value.

Deoxidizer titration range: Within ± 1 ml of the value which gives the desired results.

Etch Rate:
The most reliable method to control the fluoride content of the bath is to control the etch rate. The etch rate is determined by immersing a panel for a specific length of time and measuring the mass loss.

$$\text{Etch Rate} = \frac{(I - F)\ (Th)\ 30}{(I)\ (I.T.)} \quad \text{(mils/surface/hour)}$$

I = Initial mass (grams)
F = Final mass (grams)
Th = Initial Thickness (mils)
I.T. = Immersion Time (minutes)

A 2024 Clad panel should exhibit an etch rate of 0.1 - 0.4 mils/side/hour. For most applications an etch rate of 0.15-0.20 is best. Once an optimum etch rate is established, it should be maintained at approximately ± .025.

To raise the etch rate by approximately 0.100 mils/side/hour, add 0.9 gallon of Additive A per 100 gallons of bath.

Replenishment:
Mechanical Loss:
Whenever a portion of the bath is discarded or otherwise lost, the volume should be restored with the same proportion of chemicals and water as used in the original bath makeup.

Normally, the Deoxidizer Titration is maintained by additions of Deoxidizer 16 Replenisher. However, due to variations in dragout as compared to the amount of chemicals consumed by the deoxidizing process, periodic additions of Deoxidizer 6 Makeup may be necessary. The most practical means of determining the required replenisher is to monitor the etch rate of the bath as well as the titrations. The following table gives the necessary additions of the respective additives.

To raise the Deoxidizer Titration by one ml, add	Etch rate of 2024 clad alloy will increase by approximately
42 fl oz (0.33 gal)/100 gal DX16,	.0175 mils/surface/hour
or 42 fl oz (0.33 gal)/100 gal DX6,	.0044 mils/surface/hour

In cases where the etch rate needs to be raised when the deoxidizer titration is within range, an addition of 8.8 fl oz (0.068 gal) Additive A/100 gals of bath will increase the etch rate (of 2024 clad alloy) by 0.01 mils/surface/hour. Note: These replenishment values regarding etch rate are applicable to baths which have been in use. For relatively new baths, the required amount of Additive A may be less than the amount indicated above.

Important: Note that etch rates are given for clad 2024 alloy in a still (non-agitated bath). Other alloys, such as bare 2024, can have etch rates up to 50% higher than the values listed. Excessive agitation in the deoxidizer bath should be avoided as the etch rate will increase with increased solution flow rate around the parts.

DE7-17 Toner can be added to prolong bath life. The Deoxidizer DE7-17 Toner can be added whenever an addition of Deoxidizer 16 is needed to raise the Deoxidizer Titration of the bath. For every 100 gallons of Deoxidizer 16 added, add approximately 4.4 gallons of Deoxidizer DE7-17 Toner. In unusually extreme cases where dissolved copper builds up in the bath despite routine Deoxidizer DE7-17 Toner additions, the Deoxidizer DE7-17 Toner can be added separately. An addition of 0.15 gallons of Deoxidizer DE7-17 Toner per 100 gallons of bath will reduce the copper content by approximately 70 ppm.

Alternatively, if instrumentation is available to routinely monitor the copper content of the Deoxidizer 16 bath, the Deoxidizer DE7-17 Toner can be added only as necessary to maintain the copper content below 200 ppm, using the above addition rate of Deoxidizer DE7-17 Toner. Excessive additions of Deoxidizer DE7-17 Toner are not recommended.

Reaction Product Titration:
As aluminum is processed, reaction products will gradually accumulate unless there is sufficient drag-out of the bath during processing.

Pipet a 5 ml sample into an iodimetric flask and dilute to approximately 100 ml with water. Add 2 to 4 ml of Auxiliary Test Solution 53 and mix well. Add 7 to 8 ml of Reagent Solution 46. Add several boiling chips or glass beads and bring to a boil. (NOTE: The glass beads may be reused.) Boil for about 20 minutes and then cool to room temperature.

NOTE: The solution should be yellow in color. If it is green, after cooling repeat the addition of Reagent Solution 46 and again boil for about 20 minutes and cool to room temperature.

Add about 15 ml of Reagent Solution 49. Add 1 teaspoonful (approximately two grams) of Reagent 2 and agitate the solution until the Reagent 2 is dissolved.

After the sample has settled for approximately one minute, titrate with Titrating Solution 104 until a straw color is obtained. Do not rezero the buret. Add several ml of Indicator 10, stir, and continue the titration until the blue-black color disappears. The ml of Titrating Solution 104 used is the Reaction Product Titration.

Control of Reaction Products:
Determine the Reaction Product Level as shown in the equation:

Reaction Product Level = Reaction Product Titration (RT)
minus Deoxidizer Titration (DT)

When using the DEOXIDIZER 6/16 process without the addition of Deoxidizer 7-17 Toner, the maximum RT-DT value is 30 points, above which part of the bath volume should be decanted.

When using the DEOXIDIZER 6/16 process with the addition of Deoxidizer 7-17 Toner, the maximum RT-DT value is 70 points above which part of the bath volume should be decanted.

Record the number of days required for the Reaction Titration minus the Reaction Product Level (RT-DT) to reach a value of 70.0.

Divide the number of gallons in the bath by the number of days found above. This value is the number of gallons of bath which should be drawn off daily to keep the level of reaction products below 70.0.

Example: A 500 gallon bath requires 20 production days to reach an RT-DT value of 70.0. Therefore, 25 gallons per day should be withdrawn from the bath (500 divided by 20 = 25). This amount should be prorated per processing hour, i.e. for an 8 hour day, 3.1 gallons should be removed per hour (25 / 8 = 3.1 gal/hr).

9. After Treatment:

After the work has been treated in the DEOXIDIZER 6/16-Acid bath, it must be thoroughly rinsed with cold or hot water (a cold water rinse results in lower surface resistance). It is then ready for further processing. If the work, after rinsing, is to be welded, it should be dried and handled carefully to avoid soiling the surface. If the work is to be treated with ALODINE® coating chemicals, it is not necessary to dry after rinsing.

10. Storage Requirements:

Deoxidizer 6 Makeup and Deoxidizer 16 Replenisher will freeze at 19° Fahrenheit and 0° Fahrenheit, respectively. Toner DE7-17 will freeze at 25° Fahrenheit. Freezing is not detrimental to the products. Should they freeze, simply thaw them in a warm place and stir prior to use. The active components of Additive A will begin to precipitate below 30° Fahrenheit. If this should occur, move the chemical to a warm place and stir until fully dissolved. It is recommended that the products be stored indoors in a cool, dry place apart from combustible or oxidizable materials.

11. Waste Disposal Information:

Applicable regulations covering disposal and discharge of chemicals should be consulted and followed.

Disposal information for Deoxidizer 6 Makeup, Deoxidizer 16 Replenishing, Toner DE7-17, and Additive A is given on the Material Safety Data Sheets for each product. The processing bath is acidic and contains nitric or sulfuric acid, hexavalent and trivalent chromium and fluoride. Waste treatment and neutralization will be required before discharging.

The processing bath and sludge can contain ingredients other than those present in the chemical as supplied and analysis of the solution and/or sludge may be required prior to disposal.

12. Precautionary Information:

When handling the chemical products used in this process, the first aid and handling recommendations on the Material Safety Data Sheet for each product should be read, understood and followed.

The processing bath is acidic and may cause irritation or possibly burns to skin and eyes. The bath solution can be toxic if mist or spray droplets are breathed. Avoid contact and avoid breathing mist or spray droplets.

The processing bath contains chromic acid in excess of 0.1% when prepared as recommended. The following statement or one similar to it should be included as part of the process tank labeling.

"Possible cancer hazard based on tests with laboratory animals.
Overexposure may create cancer risk."

Refer to Material Safety Data Sheets for additional information.

Testing Reagents and Apparatus
(Order only those items which are not already on hand)

Code	Quantity	Item
205185	1 qt	Auxiliary Test Solution 53 (50% caustic soda)
205246	1 qt	Reagent Solution 46 (30% hydrogen peroxide)
205402	3*	Beaker, 250-ml, Glass
205999	1	Bung Wrench
205700	1	Buret Assembly, 25-ml Automatic, Glass
205851	1	Cylinder, graduated, 25-ml, Glass
205853	1	Cylinder, graduated, 100-ml, Glass
205897	3*	Flasks, iodimetric, 250-ml, Glass
205994	1 box	Glass beads, 4mm
205591	2	Indicator Bottle, clear plastic, 4 oz.
205002	1 qt	Indicator 2 (Bromcresol Green)
205010 .	1 qt	Indicator 10 (Soluble Starch)
205941	2*	Pipet, 2-ml, Glass
205942	2*	Pipet, 5-ml, Glass
205947	1	Pipet Filler
205082	1 lb	Reagent 2 (Potassium Iodide)
205249	5 pint	Reagent Solution 49 (CP hydrochloric acid)
205984	1	Pocket Thermometer (0-220°F)
205289	1 gal	Titrating Solution 89 (1.0 N Sodium Hydroxide)
205104	1 gal	Titrating Solution 104 (0.1 N Sodium Thiosulfate)

* Includes one more then actually required, to allow for possible breakage,

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Revision History:

Date Created: 17-08-1998 21:33 **Last Edited:**
Created By: Lisa Yurkie **Edited By:**

Form Revised 16 May 1997