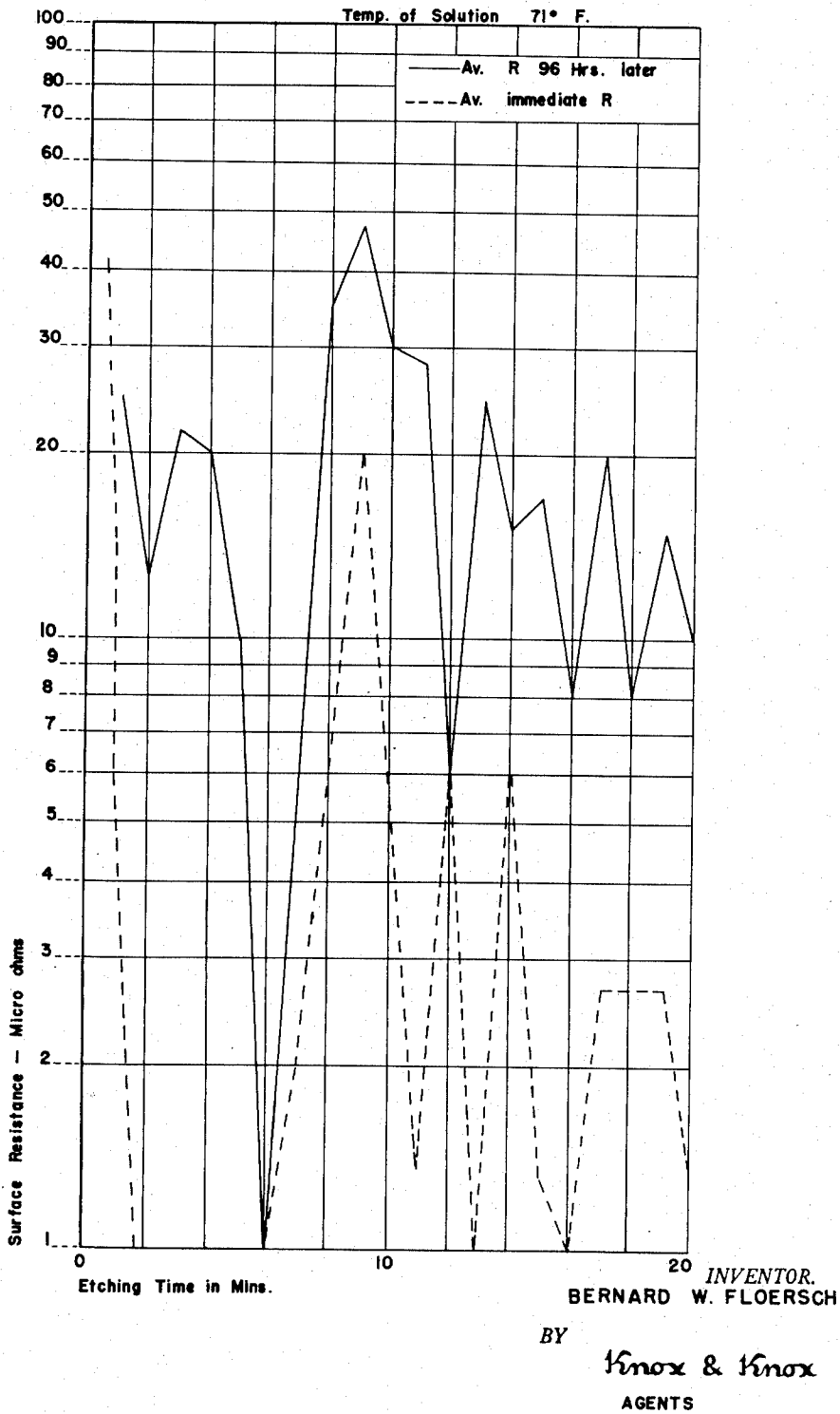


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COMPOUNDED FLUOBORATE CHEMICAL
SURFACE TREATMENT OF ALUMINUM
AND ALUMINUM ALLOYS
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COMPOUNDED FLUOBORATE CHEMICAL
SURFACE TREATMENT OF ALUMINUM
AND ALUMINUM ALLOYSBernard W. Floersch, San Diego, Calif., assignor
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10 Claims. (Cl. 41-42)

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This invention relates to the chemical surface treatment of aluminum and aluminum alloy material to remove oxides and to remove the lead and zinc contamination encountered in drop hammer forming as a result of the use of lead punches and zinc alloy dies in such forming.

The primary object of this invention is to remove the oxides and contamination in order that the metal surface be prepared for electrical resistance welding and possibly other processes.

Another object of this invention is to provide a process which overcomes the defects encountered in the use of presently known processes such as the use of fluosilicic acid used alone or with wetting agents. By way of explanation, the defects are principally over-etching, non-uniformly low surface resistance, and short holding time. With regard to the over-etching, it is pointed out that this over-etching tends to be selective so that a non-uniform surface is the result. It is true that a more uniform surface is attained when the etching is limited to a time period of 12 to 15 minutes, but this time is often insufficient to complete the removal of the lead and zinc from the surface of the material. The high resistance, or non-uniformly low resistance, is the result of the incomplete removal of the contaminating metal. The holding time is that period during which there is no change in the surface resistance of aluminum after the same is removed from the etching solution, and it has been found that this holding time in the case of etches heretofore proposed, at least in the case of the fluosilicic acid, rarely exceeds 12 hours. The instant invention overcomes each of the said defects. In addition, the present invention overcomes the tendency for a black deposit of finely divided iron to form on the material. Certain proportions of the copper in the aluminum alloys results in the deposition of copper on the surface of the material due to galvanic action, and when fluosilicic acid is used the deposition of iron and copper, generally termed smutting, is a serious problem, whereas with the present invention such smutting is completely avoided.

Another important object of this invention is to provide a process which is very speedy, a time lapse of two minutes being usually sufficient for a satisfactory immersion. In this regard, it is also very important that the desired low surface resistance of approximately zero microhms is attained in two to four minutes and again after a twenty minute immersion time. In general, commercial processes now used exhibit an increase in surface resistance after the optimum immersion

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time has been exceeded. However, under ordinary circumstances the resistance will not increase rapidly after a twenty minute period in the use of the presently disclosed process.

5 In the drawing accompanying this specification and constituting a part of this disclosure, the single figure is a graph showing the surface resistance of material plotted against etching time.

In brief, the etching compound is comprised of only three active substances, two de-oxidizers and a mild oxidant. Fluoboric acid, provided by adding a fluoborate, is the main etchant, nitric acid is the secondary de-oxidant, and chromic acid is the mild oxidant and metal complexer. The criteria used in determining the proper composition of the present etch may be thought of as the zero surface resistance to be achieved. The reason for this is that weldability or good penetration and shear strength is dependent on low surface resistance. The complete procedure for treatment of 61S aluminum consists of a five-minute treatment in alkaline precleaning solution, followed by a two to four-minute etch using the etching compound disclosed immediately below, and then followed by a one-minute post-etch in 1.8% solution, by weight, of fluosilicic acid. The etch consists of the following ingredients in the indicated percentages by weight:

	Per cent by weight
30 Chromic acid.....	0.1 to 1.0
A source of fluoborate anion.....	0.5 to 6.0
Nitric acid.....	2.0 to 12.0

The foregoing useful ranges of the components of the etch are obviously not particularly limiting, and this fact will be recognized as important since it means that the etching fluid can be used for a considerable time without constant checking of the percentage composition thereof. Furthermore, the immersion time is not unduly critical and the material may be removed after two or three minutes immersion or after a twenty minute period if more convenient. This is true because the surface resistance does not change materially during the longer period of immersion and no smutting or plating with copper is experienced.

The source of fluoborate anion may be fluoboric acid, ammonium fluoborate, a fluoborate salt of ammonia or alkali metal such as sodium fluoborate, the last mentioned being possibly preferable in most instances. The graph in the accompanying drawing will be self-explanatory, the surface resistance being reduced to approximately zero microhms in two to four minutes and again reaching this low resistance after an immersion

period of approximately twenty minutes. It is noteworthy that the time of immersion can be extended considerably beyond the twenty minute period, shown on the graph, without an increase in surface resistance. After 96 hours the surface resistance does not exceed 50 microhms. Finally, it may be mentioned that the instant process was primarily developed for preparation of aluminum and aluminum alloy material for resistance welding, but it is conceivable that other uses may be found for the process.

In summary, the etch produces a uniform, low resistance surface faster than most etches, is unaffected by small temperature changes, at least between the limits of 65° F. to 80° F., retards over-etching, possesses a long holding time, and prevents the smutting or plating of copper and iron on the surface of the material.

Further description would appear to be unnecessary.

It is understood that minor variation from the form of the invention disclosed herein may be made without departure from the spirit and scope of the invention, and that the specification and drawing are to be considered as merely illustrative rather than limiting.

I claim:

1. A process for surface treatment of aluminum and aluminum alloy material to remove oxides and surface contamination encountered in forming operations, comprising pre-cleaning the material with an alkaline solution, then immersing the material in an etch consisting of 0.1% to 1.0% of chromic acid, 0.5% to 6.0% of sodium fluoborate and 2.0% to 12.0% of nitric acid, said percentages being by weight, and then post-etching the material with a solution of fluosilicic acid.

2. A process according to claim 1 wherein the pre-cleaning step is of approximately five minutes duration and the post-etching step is of approximately one minute duration.

3. A process according to claim 2 wherein the percentage by weight of the fluosilicic acid in said post-etching solution is 1.8%.

4. A process according to claim 1 wherein the time of said immersion in the etch is of the order of three minutes.

5. A process for surface treatment of aluminum and aluminum alloy material to remove oxides

and surface contamination encountered in forming operations, comprising immersing the material in an etch consisting of 0.1 to 1.0% of chromic acid, 0.5 to 6.0% of sodium fluoborate and 2.0 to 12.0% of nitric acid, said percentages being by weight.

6. A process of preparing aluminum and aluminum alloy material for welding, comprising immersing the material in an etch consisting of 0.1 to 1.0% chromic acid, 0.5 to 6.0% of a source of fluoborate anion and 2.0 to 12.0% of nitric acid, said percentages being by weight.

7. A process of preparing aluminum and aluminum alloy material for welding, comprising immersing the material in an etch consisting of 0.1 to 1.0% chromic acid, 0.5 to 6.0% of a source of fluoborate anion and 2.0 to 12.0% of nitric acid, said percentages being by weight, for approximately three minutes.

8. A process of preparing aluminum and aluminum alloy material for welding, comprising pre-cleaning the material in an alkaline solution, then immersing the material in an etch consisting of 0.1 to 1.0% chromic acid, 0.5 to 6.0% of a source of fluoborate anion and 2.0 to 12.0% of nitric acid, said percentages being by weight, and then post-etching the material with a solution of fluosilicic acid.

9. A process of preparing aluminum and aluminum alloy material for welding, comprising immersing the material in an etch consisting of 0.1 to 1.0% chromic acid, 0.5 to 6.0% of sodium fluoborate and 2.0 to 12.0% of nitric acid, said percentages being by weight.

10. A process of preparing aluminum and aluminum alloy material for welding, comprising immersing the material in an etch consisting of 0.1 to 1.0% chromic acid, 0.5 to 6.0% of sodium fluoborate and 2.0 to 12.0% of nitric acid, said percentages being by weight, for at least two minutes at ambient temperature.

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