HEAT TREATMENT OF ELECTROLESS NICKEL DEPOSITS

General

Any heat treatment of electroless nickel deposits is preferably carried out in an atmosphere that is slightly reducing. This will maintain a bright plate, free from discoloring oxide film. Heat treated parts should never be exposed to the air until the temperature is below 750°F (400°C). One hour at 750°F (400°C) will produce maximum hardness. Two hours at 750°F (400°C) will cause the deposit to lose maximum hardness due to annealing.

To avoid unnecessary tying-up of atmosphere furnaces during cooling periods, it is generally advisable to construct sealed retorts in which the work to be treated can be loaded and purged outside the furnace, then heat treated with a constant flow of the reducing atmosphere, removed from the furnace at the completion of the proper heat treat cycle, and cooled, still under reducing atmosphere, while a second retort is in the furnace undergoing heat treatment.

An atmosphere which has been found to be very suitable for this type of heat treatment is one consisting of 10% hydrogen and 90% nitrogen. This gives a perfectly safe atmosphere with slightly reducing characteristics. It is nonexplosive under any conditions. Such an atmosphere can be obtained from the mixing of bottled gases or, in slightly different proportions, from cracking natural gas in one of the commercial atmosphere generators. It is also possible to purchase a generator which will crack ammonia, giving a gaseous mixture of nitrogen and hydrogen. This mixture contains a higher percentage of hydrogen and is more dangerous to handle as it can become explosive under certain conditions.

Salt bath heat treating has been tried and found to work. This method of heat treating has been found particularly useful when it is desired to increase the hardness of the coating on an aluminum base without affecting the heat treat characteristics of the base aluminum. It has been found that the deposit skin can be hardened in a matter of seconds, such that the heat does not penetrate to the aluminum base sufficiently to affect its properties. However, the corrosion resistance properties of the coating are affected adversely by this treatment.

In general, furnace heat treating is preferable to the salt bath heat treating because the latter requires extremely close control as to the time and temperature, and also does not lend itself to a large scale operation.

Increasing Hardness

When as plated electroless nickel alloys are heated at 750°F (400°C) for a suitable time period (function of the applied temperature), the structure recrystallizes to a mixture of nickel-phosphorous solid solution and a nickel phosphide. Ni_3P, in particulate dispersion. The fineness of the dispersion decreases (or the mean particle size increases) with increasing annealing temperature and/or time). The initial rise in hardness is associated with the migration of the nickel phosphide from the homogeneous amorphous state to the crystalline phase mixture. Between 400° and 750°F (200° and 400°C) hardness (measured at room temperature) can be varied from the as-plated condition (about 500 Vickers or 49 Rockwell C) to a maximum of over 1000 Vickers (approximately 70 Rockwell C) (Figure 1). at 750°F (400°C) in an inert atmosphere, the maximum hardness is obtained in less than 10 minutes, and an increase in the time of baking, up to one hour, has no noticeable effect.

If heat treatment is carried on at temperatures above 705°F (400°C), hardness values will decrease until, at approximately 1350°F (730°C), they reach again the as-plated condition, and thereafter apparently level out. Figure 2 shows that increased hardness can be attained at lower heat treating temperatures for longer periods.

Decreasing Brittleness

At maximum hardness values, electroless nickel deposits are brittle. In those cases where parts are subject to high loads or the possibility of damage from impact, the heat treatment should be carried out at a higher temperature (or for much longer time periods), i.e., beyond the maximum of the curve (right side, Figure 1), preferably using an inert or slightly reducing atmosphere. As already mentioned, this will result in a decrease of the hardness (through an increase in particle size of the nickel phosphide crystals). Thus, while one hour at 750°F (400°C) resulted in a Vickers Hardness Number 1195 in one careful experiment, the corresponding figures for higher annealing temperatures were as follows:

Temperature °F	Temperature °C	Vickers Hardness Number	Rockwell C
932	500	980	68
1112	600	739	62
1292	700	665	58.5
1472	800	544	52

For improved resistance to plastic strain, heat treatment of 932° to 1112°F (500° to 600°C) or above, should be used if hardness is not important.

Relief of Hydrogen Embrittlement (Alloy Steels)

Appreciable quantities of hydrogen are absorbed, both by the nickel deposit and by the base metal during pickling and plating (electroless as well as electrodeposition) When the substrate is steel, the effect of absorbed or dissolved hydrogen will seriously affect certain mechanical properties. Hydrogen in excess of two parts per million (roughly 1 cc of H_2 (S.T.P.) per 100 g of iron), and especially in the range of 10 ppm, greatly reduces the tensile ductility of all steels.

The rate of evolution of absorbed hydrogen is primarily governed by the diffusion rate of the gas through the steel and the coating, and is a direct function of temperature.

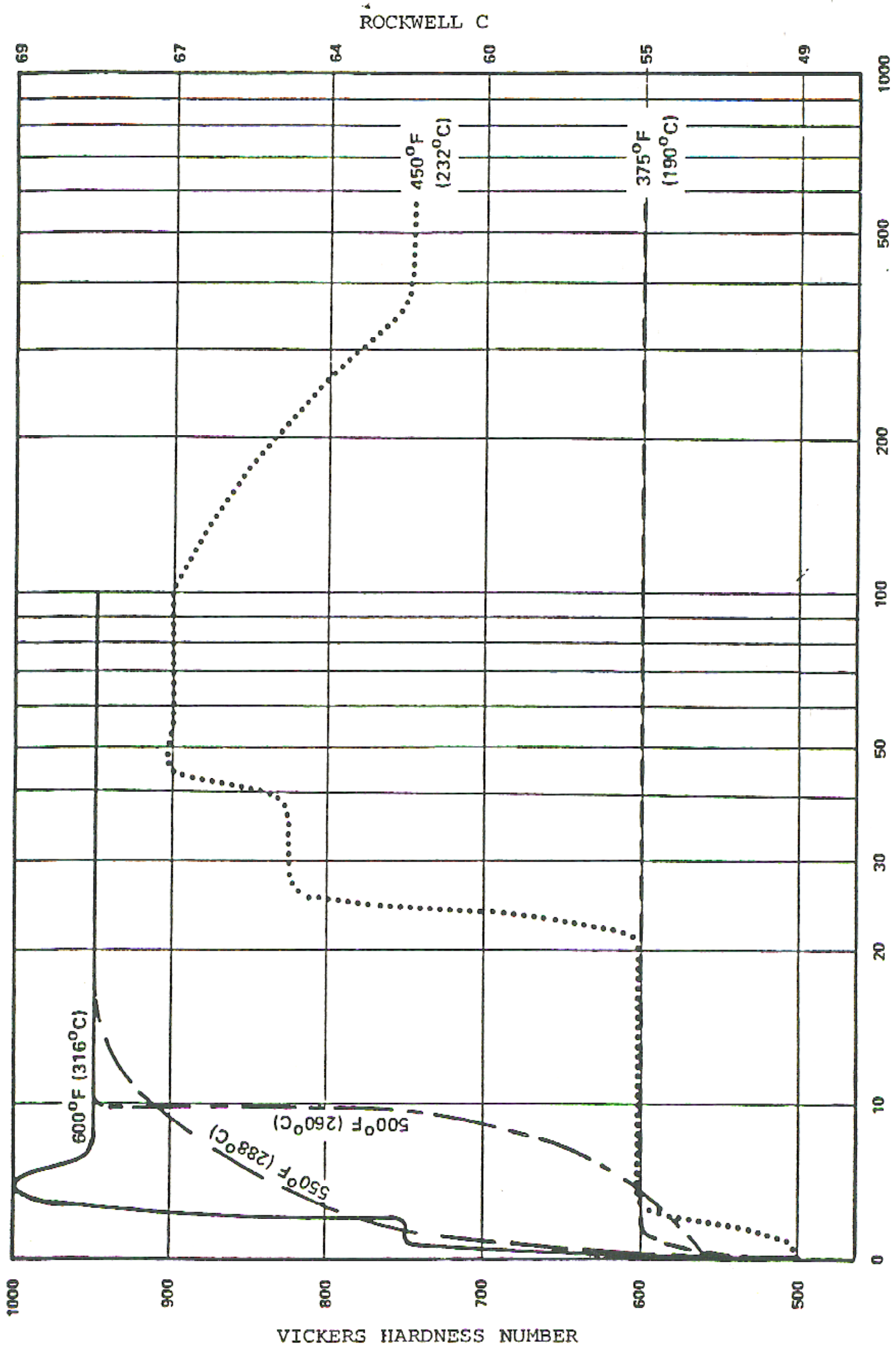

Figure 2. Heat Treating Temperature And Time Vs. Hardness Of Electroless Nickel

Hot Hardness

At elevated temperatures, the hardness of nickel-phosphorus coatings decreases rapidly, as shown by the following data:

Test Temperature °F	Test Temperature °C	Vickers Hardness Number	Rockwell C	Strain To Cause Cracks In Deposit
77	25	500	49	2.2% elongation
750	400	320	32	----------
900	483	230	18	2.5% elongation
980	527	160	--	4.0% elongation
1100	594	Too soft for measurement		----------

Improvement of Adhesion

The adhesion of electroless nickel deposits on some substrates is improved by baking at relatively low temperatures in a natural (air) atmosphere from 250° to 500°F (120° to 260°C), or in an inert or slightly reducing gaseous medium from 500° to above 900°F (260° to above 480°C). This post-treatment is strongly suggested for electroless nickel plated aluminum, titanium and alloy steels. It is generally recommended for most base materials. However, some aluminum alloys are adversely affected by heat treatment in excess of 250°F (120°C). Therefore, it becomes important to know the physical characteristics and annealing properties of these alloy so that the baking temperature can be maintained just under that which would cause a lowering of tensile strength. (In such cases, the baking time would be increased accordingly.) The usual baking time is one hour for a coating thickness of 2 mils or less, and 4 hours for thicknesses from 2 mils up to 5 mils. The recommended temperatures for adhesion improvement are as follows:

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