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# Cladding Metals by Continuous Strip Rolling in Vacuum

By Robert Blickensderfer

UNITED STATES DEPARTMENT OF THE INTERIOR

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# CLADDING METALS BY CONTINUOUS STRIP ROLLING IN VACUUM

by

Robert Blickensderfer [1]

ABSTRACT

Equipment and procedure for the continuous roll bonding of metal strips are described. The reels, mill rolls, and furnace were located within a vacuum chamber capable of operation at a residual gas pressure of 1 x $10^{-4}$ to 1 x $10^{-5}$ torr. Several metals were successfully clad at rolling temperatures of 930° to 1,050° C in lengths up to 120 ft. The bimetals that were clad included stainless steel to carbon steel, molybdenum and nickel to stainless steel and carbon steel, and titanium to carbon steel. Bend tests of bond integrity are described, and microstructures of the interfaces are presented. The amounts of brittle intermetallic phases formed at the interface are minimized by this cladding method.

## INTRODUCTION

The increasing need to conserve the dwindling resources of certain scarce metals justifies investigating some of the less common and more technical methods for achieving conservation. One of the more technical methods for stretching the use of scarce metals is to clad an abundant base metal with the scarce metal.

Present technology includes several batch processes for cladding that are marginally economical; for example, explosive cladding, weld overlaying, and sealed-pack hot-rolled cladding. Development of a continuous process for cladding was emphasized in the work reported here because continuous processes in general have considerable economic advantage over batch processes.

The U.S. Department of the Interior, Bureau of Mines investigated the potential of continuous strip cladding by vacuum rolling. The vacuum is necessary to prevent oxidation of the metals during heating because oxide layers inhibit metallic bonding between the cladding and the base metal. Numerous advantages and disadvantages of vacuum rolling are given by Beall (1).[2] Another advantage, learned from our recent work on one-pass vacuum rolling(2),

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[1] Metallurgist, Albany Research Center, Bureau of Mines, Albany, Oreg.
[2] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

was the small amount of diffusion that occurs across the interface. This prevents the formation of brittle intermetallic or carbide phases. Nevertheless, the high technology required to operate rolling equipment inside a vacuum chamber is a deterrent to any vacuum rolling process.

The literature on vacuum rolling (1, 3-4) indicates that most of the work was on a small laboratory scale. One exception was a series of rolling mills in vacuum in the U.S.S.R. (5) capable of rolling billets into tubes. The other exception, and the only description of continuous strip cladding, is in a U.S. patent by Ulam (6), in which inert gas or vacuum was specified for cladding a stainless steel strip to an aluminum strip.

## CONTINUOUS STRIP ROLLING

### Equipment

Establishing the equipment required to continuously roll and bond two strips of metal in vacuum was a major part of the research effort. A schematic of the general arrangement of the equipment is shown in figure 1. Figure 2 shows the rolling mill chamber with the door open to reveal the payoff reels. The rolling mill, furnace, and takeup reel were also located within the vacuum chamber, which had a volume of approximately 55 $ft^3$ (1,560 $\ell$). Three diffusion pumps with a total pumping rate of 53 $ft^3$/sec (1,500 $\ell$/sec) were used to maintain vacuum during rolling.

The rolling mill, rated at 175,000 lb force, used two hardened steel-alloy rolls of 6-1/2-inch diameter and 8-inch face. The rolling speed could be controlled over the range of 2 to 40 ft/min. The 30-kw mill motor and drive train were located outside the vacuum chamber.

The furnace for heating the metal strip was 4 inches inside diameter by 40 inches long. It consisted of a tantalum heating element 0.005 inch thick that was split lengthwise and surrounded by four stainless steel radiation shields.

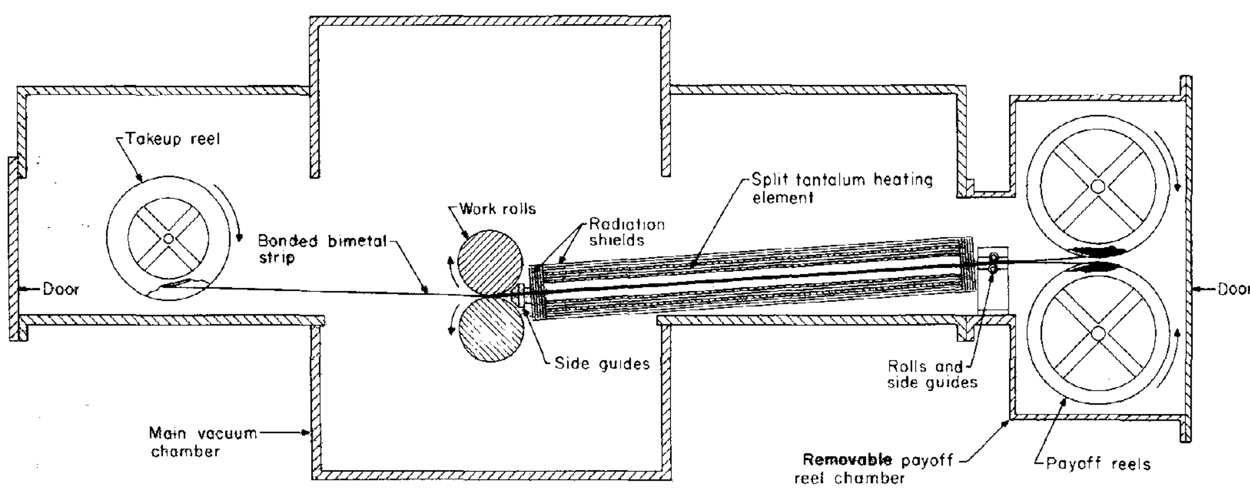


FIGURE 1. - General arrangement of continuous strip vacuum rolling mill.

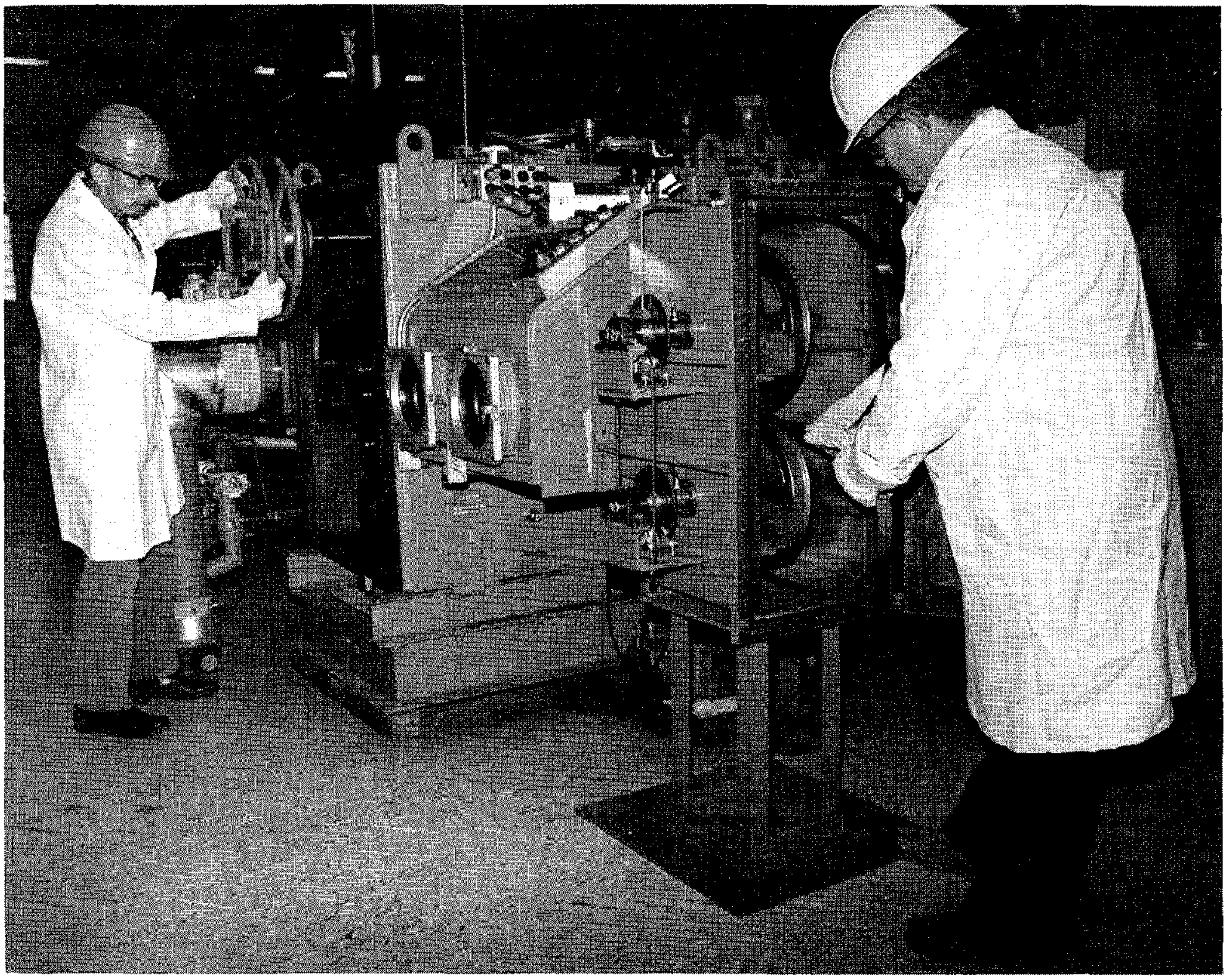

FIGURE 2. - Continuous strip vacuum rolling mill. Strips are being fed into furnace from payoff reels, at right, before closing the vacuum chamber.

Quartz spacer rings isolated the heating element electrically from the radiation shields. Electrical power was supplied by a dc welding source. Typical furnace operation required 200 amp at 25 volts or 5 kw to achieve a furnace temperature of 1,200° C. When moving metal strip was being heated to 1,000° C, the furnace power had to be increased to 10 or 15 kw depending upon strip thickness and speed. To achieve rapid response of strip temperature with changes in power setting, a furnace with low heat capacity, like this furnace, was required.

Temperatures of the strip and furnace were measured in several ways. A Pt versus Pt-10 pct Rh thermocouple, located about 6 inches from the exit end of the furnace, was used in conjunction with a digital readout to measure furnace temperature. To measure the strip rolling temperature within the small region between the furnace exit end and the mill rolls, a disappearing-element optical pyrometer, an infrared thermometer, and a rubbing contact thermocouple were tried. The latter was 200° to 400° C below the optical pyrometer reading and was inconsistent; therefore it was discontinued. The infrared thermometer gave temperatures about 200° C lower than the optical pyrometer but was consistent and could be used after calibration. The optical pyrometer readings, corrected for sight glass absorption, were considered the most accurate and were used for reporting strip temperature. The brightness temperatures were based on a 0.65-μm wavelength and an emissivity of 1.

The two payoff reels, figure 1, held the supply of the two metals to be bonded. The reel hubs were 10-inch diameter by 1-1/2 inch wide. In order to hold the tension on each strip that was required for good tracking, a disk brake was mounted on each reel shaft. The brakes, being outside the vacuum chamber, were easily adjusted to provide the desired tension. A pair of small guide rolls between the payoff reels and furnace entrance, as well as a set of guides between the furnace exit and the mill rolls, helped maintain alinement of the strips during rolling.

The takeup reel was similar to the payoff reels, except that it was power driven. A 3/4-hp dc motor was used to maintain torque on the reel to provide sufficient tension in the clad strip between the rolling mill and the takeup reel. The torque was controlled manually by adjusting the current to the reel motor.

## Materials Bonded

The metal strips used for the continuous vacuum roll bonding were obtained from commercial suppliers. All strips were 1-3/8 inches wide; the thickness ranged from 0.010 to 0.037 inch for the cladding and from 0.020 to 0.050 inch for the steels. The various strip materials are characterized in table 1.

TABLE 1. - Strip metals used for continuous roll bonding[1]

| Strip designation | Materials and grade | Thickness, inch |
|---|---|---|
| 304SS.010............. | Stainless steel, type 304.......... | 0.010 |
| 304SS.025............. | .....do............................ | .025 |
| Mo.................... | Molybdenum, commercially pure....... | .010 |
| Ni.................... | Nickel, pure, "A".................. | .010 |
| Ti.015................ | Titanium, pure, 50A................ | .015 |
| Ti.037................ | .....do............................ | .037 |
| 1018.................. | Steel, C1018....................... | .020 |
| 1030.................. | Steel, C1030....................... | .050 |

[1]All were 1-3/8 inches wide and from commercial suppliers.

The effect of surface preparation on bonding was determined in our earlier work (2) on small specimens: as long as the surface was clean and degreased, additional treatments such as abrasively sanding or chemically etching were found unnecessary. Therefore, the strips for continuous cladding were cleaned only by degreasing with Stoddard's[3] solvent, rinsing with alcohol, and wiping dry with a clean cloth. The titanium as-received appeared very clean and was used without any additional cleaning.

## Procedure

With the reel chamber (shown at the right of fig. 1) removed from the vacuum chamber, the metal strips were wound upon the payoff reels. The strips were cleaned, as described previously, while winding. After the reels were loaded with the strips, the ends of the strips were spot-welded to a stainless steel leader strip. The leader extended through the furnace and mill, and was attached to the takeup reel. Thus, tension could be maintained between each payoff reel and the takeup reel. Each drag brake was adjusted to produce a strip tensile stress of approximately one-half of the yield stress at the rolling temperature. Typical tension ranged from 25 to 80 pounds. The vacuum chamber was normally pumped down overnight before beginning a run.

To begin a run, the furnace temperature was first raised to the desired rolling temperature (about 1,000° C) for 10 minutes for outgassing. Rolling was begun with the mill opening set to produce the required reduction in thickness. During startup, control of strip temperature was difficult. After the last part of the preheated strip left the furnace, the temperature of the furnace had to be raised to 1,300° to 1,400° C to heat the moving strip. At a rolling speed of 8 ft/min, a point on the strip spent only 25 sec in the furnace.

The strip temperature during startup, with our furnace configuration, went through a cycle: The lead end of the strip, equal to the length of the furnace, was rolled at the target temperature. The strip temperature thereafter dropped markedly -- by about 300° C -- and the strips normally did not bond together. The following strip gradually got hotter until the target temperature was reached again, and bonding was achieved.

After rolling the desired length of clad strip, the furnace power was shut off, the mill rolls were opened, and the rolling mill was stopped. Several hours of cooling were allowed before opening the vacuum chamber.

## RESULTS AND DISCUSSION

Claddings of stainless steel (type 304), nickel, titanium, and molybdenum were successfully bonded to plain carbon steel by continuous rolling in a vacuum. Similarly, nickel and molybdenum were clad to stainless steel. The rolling conditions used to produce good bonding are summarized in table 2.

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[3]Reference to specific brand names is made for identification only and does not imply endorsement by the Bureau of Mines.

TABLE 2. - Rolling conditions

| Bimetal | Temperature, ° C | | Residual pressure, $10^{-4}$ torr | Reduction in thickness, pct | Rolling speed, ft/min | Length rolled, ft |
|---|---|---|---|---|---|---|
| | Furnace | Strip | | | | |
| 304SS.025/1030...... | 1,300 | 1,010 | 1 | 20 | 8 | 120 |
| 304SS.010/1018...... | 1,410 | 1,000 | 2 | 30 | 5 | 100 |
| Mo/304SS.025........ | 1,350 | 980 | 1 | 31 | 8 | 25 |
| Mo/1018............. | 1,400 | 1,050 | 2 | 25 | 5 | 25 |
| Ni/1030............. | 1,300 | 1,030 | 1 | 13 | 5 | 15 |
| Ni/304SS.025........ | 1,220 | 930 | .5 | 31 | 8 | 8 |
| Ti.015/1030......... | 1,280 | 1,035 | 1 | 20 | 5 | 52 |
| Ti.037/1030......... | 1,300 | 1,045 | | 22 | 5 | 50 |

The bonding of 304 stainless steel to either of the carbon steels was relatively easy because temperature control and oxidizing conditions were not critical. The stainless steel retained enough strength at rolling temperatures to withstand the strip tension required for good tracking. Nickel was also relatively easy to bond to steel or to the 304 stainless steel. Molybdenum proved more difficult to bond to steel because of the higher temperature required; below 1,000° C good bonding was not achieved and above 1,000° C the steel strip became too weak to be guided by the side guides. One attempt to clad molybdenum onto 304 stainless steel was successful. Bonding titanium to steel required the most strigent temperature and tension control. The very low tensile strength of titanium above 1,000° C resulted in several tensile failures of the titanium strip. Furthermore, if the strip exceeded the eutectic temperature of 1,086° C, melting occurred. One run with each of the two thicknesses of titanium bonded to 1030 steel was completed by rolling at temperatures around 1,040° C.

The integrity of the bond was determined by the peel test, bend test, and observation of the microstructure. The peel test was carried out in a very simple manner. Starting from either end of the strip, where the metals were only partially bonded, because of lower rolling temperature, the cladding was peeled back by hand. Over the region that the strips could be pulled apart with a force up to approximately 40 pounds, the bond was considered "no bond."

In regions that were not separated by the peel test, the bond might be "partial bond" or "bond." To determine which, specimens were sheared and subjected to bend tests. It should be mentioned that the shearing action region of the strip, two specimens were used -- one was bent with the cladding on the outside, the other with the cladding on the inside. The specimens were bent through an angle of 180° and then squeezed tight in a vice. If any separation of the

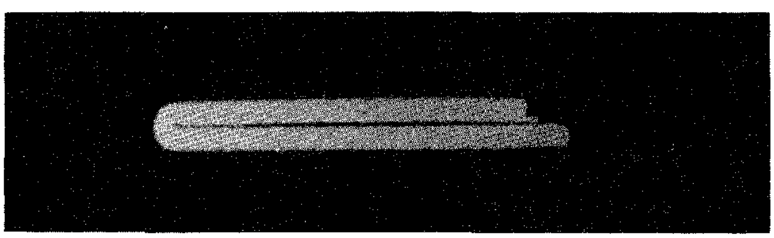

FIGURE 3. - Typical specimen after bend testing. Specimen is 304SS.010/1018. The stainless steel is on the inside of the bend (X 28).

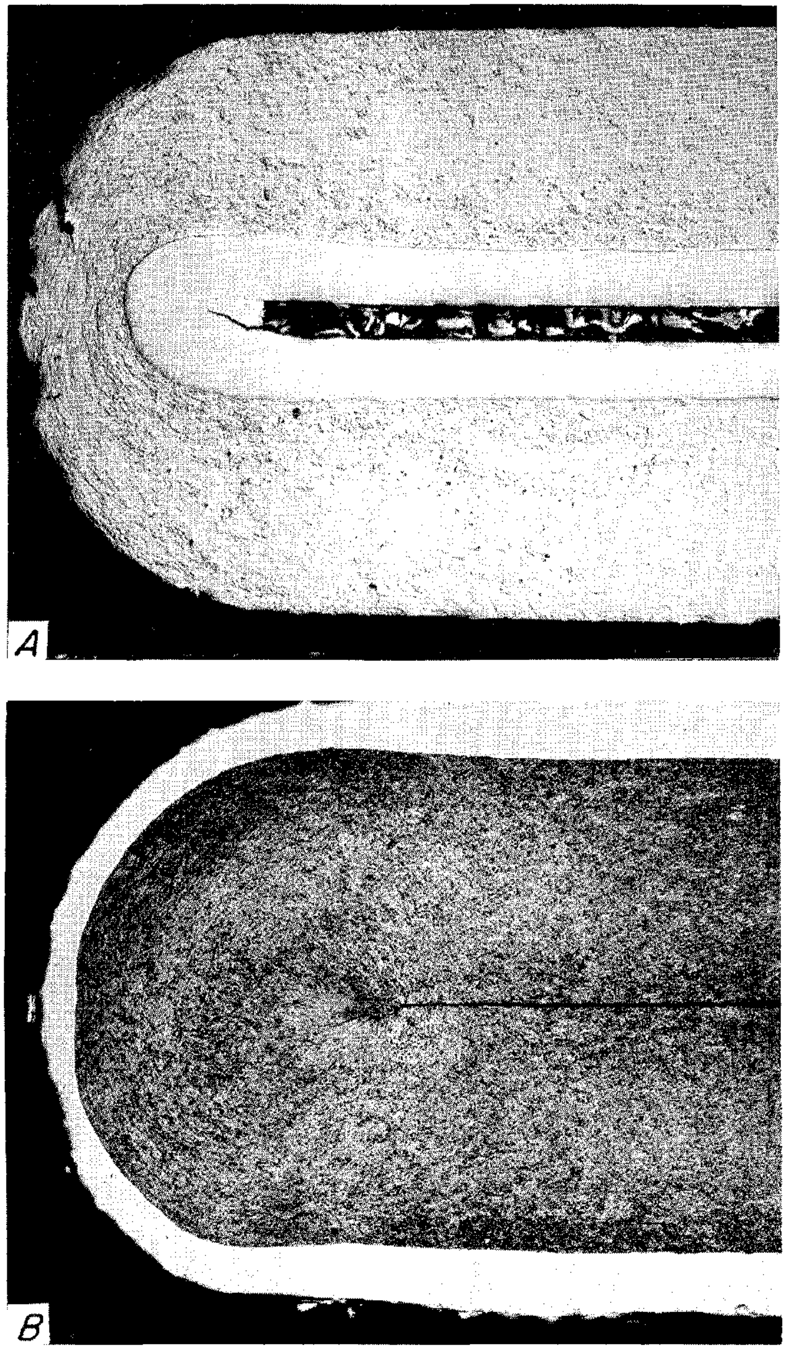

FIGURE 4. - Deformation of cladding and base after bending (X 36). Specimen is 304SS.010/1018. *A*, stainless steel cladding is on inside of bend; *B*, stainless steel cladding is on outside of bend.

cladding was detected, the bond was classified as a "partial bond." If the cladding remained attached, even though the cladding or base metal might fail in tension, the bond was judged as "bond." A macro photograph of a typical specimen after bending is shown in figure 3. Greater detail of the bend region of this same specimen, 304 SS.010/1018, is shown in figure 4. The bond between the stainless steel and the 1018 steel remained intact even though the metals were severely deformed. The validity of the bend test as a criterion for bonding was established earlier (2) by shear testing of claddings.

Microstructures of several other bimetals are shown in figures 5-9. The interfaces are seen to be quite clean and relatively free of impurities. During chemical etching the interface was attacked about the same degree as the grain boundaries. Analysis by scanning electron microscope and electron microprobe showed that very little diffusion occurred across the interface-- on the order of 0.5 to 1 μm. It was concluded that a metallurgical bond was achieved by atomic bonding across the interface similar to the bonding across a grain boundary. No intermetallic phases were detected in the specimen.

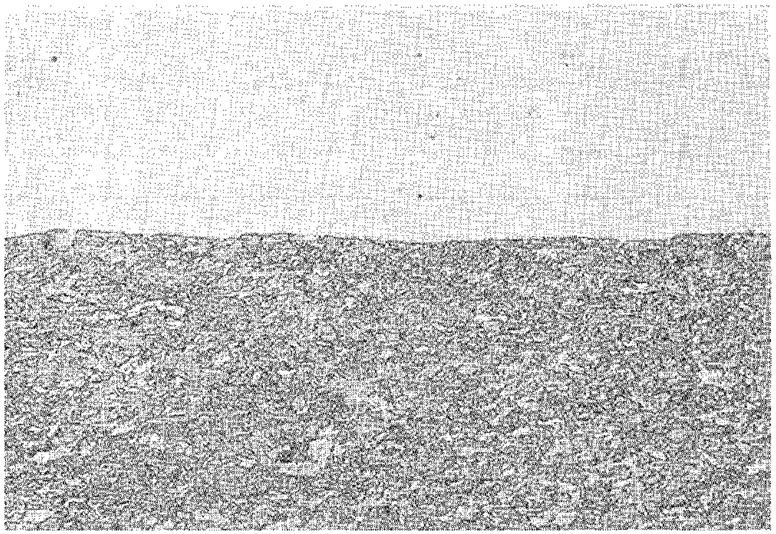

FIGURE 5. - Specimen 304SS.010/1018 (X 300).

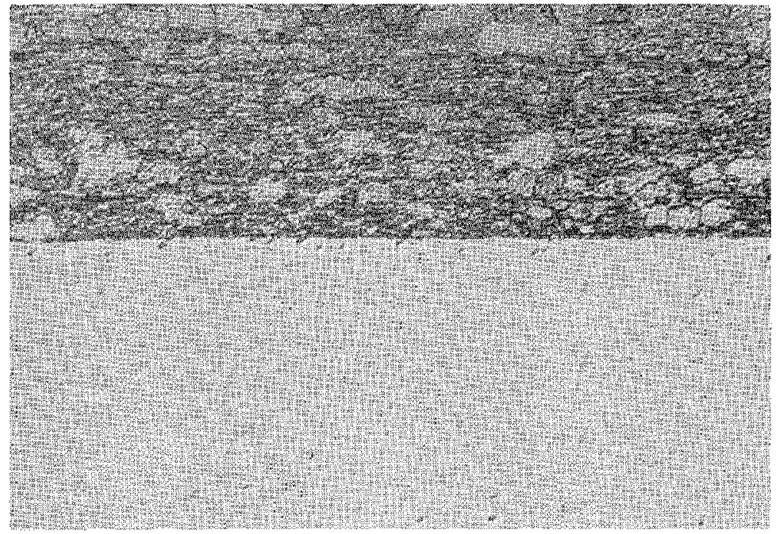

FIGURE 6. - Specimen Mo/304SS.025 (X 300).

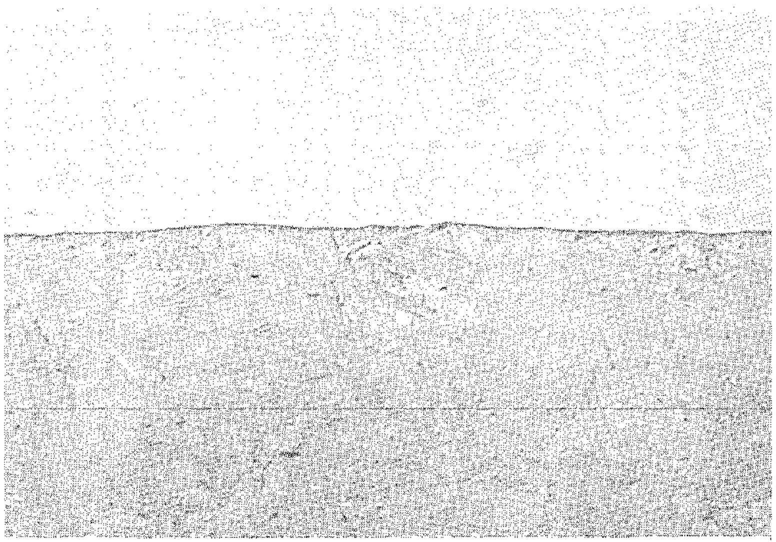

FIGURE 7. - Specimen Mo/1018 (X 300).

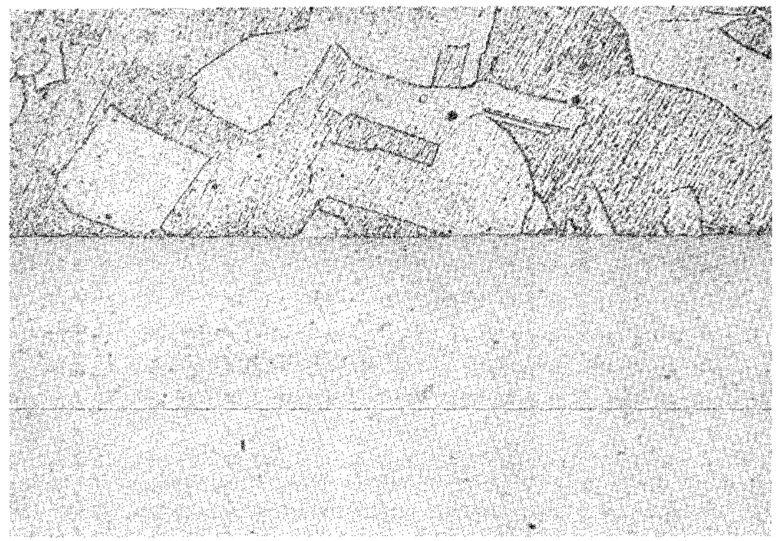

FIGURE 8. - Specimen Ni/304SS.025 (X 300).

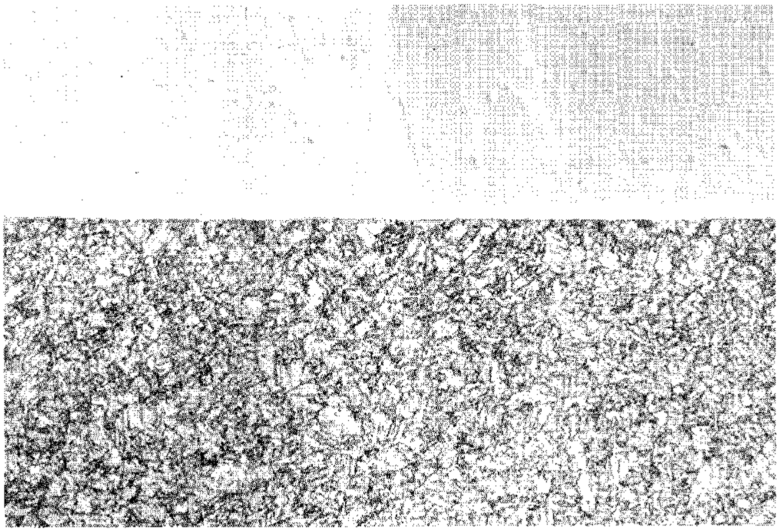

FIGURE 9.- Specimen Ti.015/1030 (X 300).

Preventing the formation of intermetallic phases gives our one-pass vacuum bonding a distinct advantage. Cladding methods that require longer time at elevated temperature can result in the formation of brittle intermetallic phases at the interface. For example, prolonged hot rolling of titanium-iron bimetal tends to form $Fe_2Ti$ and FeTi brittle phases, and subsequent deformation of the product results in failure at the interface. Again, in bonds between carbon steel and stainless steel, the diffusion of carbon into the stainless steel grain boundaries can result in excessive chromium carbide formation and consequent embrittlement.

## PROBLEMS AND RECOMMENDATIONS

The major problem encountered in continuous strip cladding was the variable strip temperature during startup. The problem occurred while making the transition from the stagnant nonmoving condition to the steady-state rolling condition. The temperature variation itself was not a serious problem, but it sometimes led to one; namely, a cobble. During the low-temperature period of nonbonding, the strips elongated to different lengths because of their differences in flow stress. Consequently, a loop of strip developed between the mill rolls and takeup reel that often resulted in a cobble.

Conceptually, there are at least three ways of reducing the startup temperature cycle problem. One way would be to make the furnace much longer and thereby to reduce the temperature differential between the furnace and the strip. That is, as the furnace length approached infinity, the temperature differential would approach zero. A very long furnace would require little temperature change between startup and the steady-state rolling condition.

Another way of reducing the temperature cycle problem would be to use a variable-temperature zone furnace, or separate furnaces in series. The strip could then be preheated with a temperature gradient, and as rolling began, the power to all zones could be increased. The final heating might be better done with an induction coil in order to minimize the length of hottest zone and its response time.

A third approach of reducing temperature fluctuation would be to fully instrument and automate the entire system. Automation, in conjunction with one of the above recommendations, would be desired for production rolling anyway. The furnace temperature, strip temperature at furnace exit, strip entry tension, rolling mill speed, and winding tension would be monitored and controlled. A programed microprocessor could be used to optimize the rolling conditions.

Another problem encountered in the course of the investigation was a tendency for thin cladding to become misalined at the rolling mill entry. From earlier experiments with bimetal specimens we found that the tail ends of two strips always tended to slip sideways during rolling. This displacement resulted from the system's seeking its lowest energy state during rolling; that is, the tail ends of the strips moved in opposite lateral directions until they could pass through the mill with the least reduction in thickness. For small specimens, the sideslip was prevented by spot-welding or reveting the tail ends together.

With the long strips and guides used in the continuous rolling set up, the alinement was maintained. However, when rolling thin cladding at high temperature, one edge of the cladding strip sometimes curled upwards along a side guide as the cladding was displaced laterally. Increasing the tension could alleviate the problem but sometimes caused tensile failure of the strip within the furnace. To assure effective guiding by the side guides, the cladding strip should be at least 0.010 inch thick -- 0.015 to 0.020 inch thick is recommended. We believe intuitively that wider sheet strips would have less tendency to sideslip and therefore would maintain alinement between the cladding and the base metal.

## SUMMARY

The feasibility of producing clad metals in strip form by continuous hot rolling in vacuum was demonstrated. Stainless steel, molybdenum, nickel, and titanium were clad to plain carbon steel; and molybdenum and nickel were clad to stainless steel. Rolling temperatures ranged from 930° to 1,050° C and residual pressures from 0.5 to $2 \times 10^{-4}$ torr.

Specimens of the clad metal strip could be bent 180° and flattened without failure of the bond. The strength of the bond is attributed to a small amount of diffusion that occurs across the interface. The microstructure of the interface was similar to a grain boundary. Brittle intermetallic phases were not detected in the interface.

The problems encountered with temperature control during startup could probably be solved in a larger scale operation with a more automated control system.

Despite the inherent difficulty of carrying out rolling operations in high vacuum, the strip cladding method should have economic potential.

## REFERENCES

1. Beall, R. A., R. B. Worthington, and R. Blickensderfer. Hot Rolling Metals in Vacuum. BuMines IC 8787, 1979, 24 pp.

2. Blickensderfer, R. Cladding of Metals to Iron by Vacuum Rolling. BuMines RI 8481, 1980 (in press).

3. Krupin, A. V. Das Walzen im Vakuum (Rolling in Vacuum). Proc. 7th Plansee Seminar on Powder Metallurgy of High Temperature Metals, Inst. of Steel and Alloy (Moscow), Rentte-Tyrol (Austria); Metallwerk Plansee, ed. by F. Benesvosky, v. 4, 1971, pp. 19.1-19.24; English translation available from Albany Research Center, Bureau of Mines, Albany, Oreg.

4. Krupin, A. V., and N. V. Yudina. Rolling of Metals and Bimetals in Vacuum. Institut Nauchnoy Informatsii. Prokatnoye i Volochetnoye Proizvodstvo, 1965. Itogi Nauki i Tekhniki, Seriya: Metallurgiya (Moscow), 1966, pp. 131-168 (in Russian): English translation by Foreign Technology Division, Wright Patterson Air Force Base, Ohio; available from National Technical Information Service, Springfield, Va., FTD-MT-24-197-67, Sept. 8, 1967, 32 pp.

5. Tsygankov, E. M., and L. A. Perfil'ev. Research at Work Laboratories and Institutes. Stal', No. 11, 1964, p. 919.

6. Ulam, J. B. Stainless Steel Clad Aluminum and Method of Making Same. U.S. Pat. No. 3,261,724, July 19, 1966, 5 pp.

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