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Summary

PS304, a plasma spray deposited solid lubricant coating developed for high temperature sliding contacts was deposited on nine different substrate metals, heat treated at 650 °C in either air or argon and subsequently tested for strength using a commercially available pull-off adhesion test. Some samples were examined metallographically to help elucidate and explain the results.

As deposited coatings exhibit pull-off strengths typically between 16 and 20 MPa with failure occurring (cohesively) within the coating. Heat treatment in argon at 650 °C results in a slight increase in coating (cohesive) strength of about 30 percent to 21 to 27 MPa. Heat treatment in air at 650 °C results in a dramatic increase in strength to over 30 MPa, exceeding the strength of the epoxy used in the pull test. Cross section metallographic analyses show that no microstructural coating changes occur following the argon heat treatments, however, exposure to air at 650 °C gives rise to the formation of a second chromium-rich phase precipitate within the PS304 NiCr constituent which provides a strengthening effect and a slight (~5 percent) coating thickness increase. Subsequent heat treatments do not result in any further coating changes. Based upon these studies, PS304 is a suitable coating for use on a wide variety of high temperature substrates and must be heat treated following deposition to enhance strength and ensure dimensional stability.

Introduction

PS304 is a plasma sprayed high temperature solid lubricant coating developed at NASA Glenn Research Center to reduce friction and wear in sliding contacts encountered in many heat engines and industrial and aerospace mechanisms (refs. 1 and 2). PS304 is made by the plasma spray deposition of a powder blend comprised of 60 wt% NiCr, 20 wt% Cr₂O₃, and 10 wt% each of BaF₂/CaF₂ eutectic and silver. Each component plays a different functional role within the composite and the PS304 coating exhibits characteristics representing the summed properties of the individual constituents (ref. 3). For instance, the NiCr acts as a binder, the Cr₂O₃ acts as a dispersed hardening agent, the BaF₂/CaF₂ is a high temperature solid lubricant and silver is a low temperature solid lubricant. As a composite, PS304 exhibits low friction and wear properties over a wide range of temperatures and is suited for deposition onto a wide variety of substrate materials. Figure 1 shows a typical PS304 cross-section. PS304 has been found to be a suitable coating for many applications including Exhaust Gas Recirculation (EGR) and Turbo-charger waste gate valve stems, foil air bearings and seals, steam turbine control valve components and bushings and shafting in heat treatment furnaces (ref. 4).

During its development, it has been observed that PS304 exhibits varying strength, measured through adhesion tests, depending upon substrate metal and thermal history. In addition, PS304 undergoes dimensional changes (thickness growth) following exposure to high temperature air environments (ref. 5). This paper reports on a systematic research effort to quantify the effects of substrate material and thermal processing on the strength, dimensional stability and microstructure of PS304. It is expected that an improved understanding of these attributes and effects will aid the transition of PS304 technology from the laboratory to commercial applications.

To study these effects, nine different alloy substrate materials were coated with PS304. Some were heat treated for 100 hr in air or argon followed by pull-type adhesion tests and subsequent metallographic analyses. A unique back-to-back specimen mounting technique was used to assess dimensional changes and standard SEM tools were employed to help understand microstructural changes within the coating.

Materials and Procedures

The primary test conducted in the program is a pull type adhesion test. Specimens were prepared by plasma-spray depositing PS304 approximately 250 μm thick onto 6 mm thick, 25 mm diameter substrate coupons. PS304 is a NiCr based solid lubricant coating developed for friction and wear reduction in high temperature sliding contacts. Table I gives the nominal composition and properties of PS304. As shown in the table, the NiCr (60 wt%) acts as a wear resistant matrix with a Cr_2O_3 (20 wt%) hardening additive. Silver (10 wt%) is added as a low temperature lubricant and $\text{BaF}_2/\text{CaF}_2$ eutectic (10 wt%) is added for high temperature lubrication. The PS304 coating and its tribological and materials properties and deposition procedures are fully described in Refs. 1 to 4.

The substrate materials considered in this study were selected because they are typical high temperature structural materials or represent a particular candidate material class (e.g., Aluminum). The substrate alloys included: the superalloys Inconel 718, Stellite 6B; the stainless steels alloy 410, alloy 304, PH 13-8Mo and Greek Ascoloy; Ti6Al-4V a common Titanium alloy and 6061 Al a high performance aluminum alloy. Table II lists the nominal composition of the substrate materials, their properties, and their general classes (e.g., precipitation hardened, austenitic, etc.).

Many of the PS304 coated specimens were subjected to various thermal processing prior to being adhesion tested. For example, some were exposed to air at 650 $^{\circ}\text{C}$ for 100 hr while others were exposed to argon gas at 650 $^{\circ}\text{C}$ for 100 hr. Control examples, which were not heat-treated, were tested in the as-deposited state for comparison. Their heat treatments were selected based upon earlier published work for PS304 deposited on 13-8Mo stainless steel which conclusively showed that brief exposure (>24 hr) to air above 550 $^{\circ}\text{C}$ results in a complete heat treatment (ref. 5). At least six specimens were prepared for each substrate and each different exposure condition (i.e., argon, air or no exposure). Additionally, one extra specimen was specially prepared for metallographic examination without undergoing an adhesion test to quantify any dimensional or microstructural changes occurring during the heat treatment. This was accomplished by slicing (using wire Electrode Discharge Machining) the coated coupon into two equal semi-circular pieces. One half was heat treated with the six full adhesion test coupons. Its mating half was reserved without heat treatment for direct comparison. Figure 2 schematically shows how these metallurgical samples were prepared. By following this process an accurate comparison of exposed and unexposed coating/substrate can be made without coating variations (i.e., thickness, voids phases) interfering in the measurements. Since earlier work (ref. 5) showed that the PS304 coating undergoes a significant thickness increase following heat treatment, these semi-circular samples will also assist in quantifying and understanding the cause(s) of this growth. Titanium and Aluminum are not considered high temperature alloys. For these specimens, a lower temperature heat treatment (593 $^{\circ}\text{C}$) was employed but resulted in coating delamination upon cooling. No heat-treated adhesion data is provided for these samples.

The adhesion pull test for coating bond/strength is shown in figure 3. To prepare a specimen, a PS304 coated coupon (following its heat treatment) is glued to a threaded aluminum pull stud using a two-part epoxy. The epoxy glue was allowed to set at room temperature (25 $^{\circ}\text{C}$) for 24 hr and then was oven cured at a 70 $^{\circ}\text{C}$ for 24 hr then cooled again to 25 $^{\circ}\text{C}$. The threaded stud was then screwed into the piston of the pneumatically loaded pull test fixture. The air pressure was then increased at a constant rate resulting in a loading profile of approximately 50Kgf/sec. The air pressure, and hence pull-off load, was monitored continuously during the test. Upon failure, which always occurred suddenly, the pull-off pressure was automatically recorded and the pull-off strength was calculated. Each pull-off test lasted only about 30 seconds. The data reported in this paper represents over 200 pull-off tests.

Results and Discussion

The test results are summarized in table III and depicted graphically in figures 4 to 6. Figure 4 shows the pull-off data for the as-deposited PS304 on the eight substrate alloys. There appears to be a slight variation in strength depending upon the substrate. For example, when deposited on Inconel 718 the PS304 fails at 19.9 MPa while on Aluminum failure occurs at 16.3 MPa. Although the pull-stud test is considered an adhesion test, few if any of the specimens failed at the substrate coating interface. All of the samples (except occasionally Aluminum) failed inside the PS304 coating, a cohesive failure. As such, the pull-stud data is actually measuring PS304 cohesive strength or tensile strength not substrate-coating adhesion strength.

Following a heat treatment in air (fig. 5), the PS304 becomes much stronger. In fact, almost all of the samples exhibited strengths higher than the epoxy strength. Thus the strength plot for air heat-treated PS304 is essentially a horizontal line at about 35 MPa, the epoxy cohesive strength. Various glue strength enhancements were attempted. There included different heat cure procedures and alternate surface preparations but the results were about the same. Heat treated PS304 coating exceed the strength of the epoxy. Nonetheless the results are still valuable. Any coating which is stronger in tension than the epoxy will likely withstand the stresses encountered in engineered coating applications. Figure 6 shows the effect of the argon heat treatment of PS304. The strengths are higher than the as-deposited samples but only slightly so. Also, there appears to be little effect of substrate on strength. These argon treated samples, like the air treated ones, also failed cohesively in the PS304 coating.

Early on in our research program, it was postulated that the slight substrate variation effect noted for as-deposited coatings was perhaps due to a degree of heat-treating taking place during the high temperatures resulting from plasma spray deposition process. Since Aluminum has a much higher thermal conduct with than stainless steel or Inconel 718 bulk coating temperatures would be expected during deposition. In contrast, the nickel based super alloy, Inconel 718, have relatively poor thermal conductivity leading to high surface and substrate temperatures during coating deposition. This theory would explain the differences in PS304 as-deposited strength.

To examine this theory, coating deposition experiments were performed with thermocouples embedded into the surface and near surface of the coupons. Temperatures were monitored during the Plasma-spraying process and a typical result is shown in figure 7. The jagged nature of the trace is a direct consequence of the back and forth motion of the plasma spray gun in which many passes are made to build up the thick 0.010 in. coating. Notwithstanding, the maximum temperature recorded during these trials was only about 300 °C, far lower than the 500 °C temperature needed to achieve even modest heat treatments for PS304 (ref. 5). Clearly, an “in-situ” heat treatment of PS304 was not the cause for the observed strength differences.

Another theory to explain the substrate effect is the formation of coating residual thermal stresses caused by a mismatch in thermal expansion between the coating and substrate. To test this theory, aluminum and 304 stainless steel samples were coated while providing either heating or cooling air to the back-side of the coupon. By doing this the coating can be forced into residual compression by the substrate (if it is heated) or tension (if it is cooled).

Since PS304, like most cermets, is a fairly brittle material, it will exhibit higher strength while under residual compression when subjected to a tensile load, as encountered in the pull-type adhesion tests. Table IV and figure 8 shows the results. Substrate preheating greatly increased the pull-off strength, and precooling greatly decreased the strength. Following a 24 hr heat treatment in air at 650 °C, 304 stainless steel samples which had been either preheated or precooled during the coating deposition yielded pull-off strengths essentially the same as the baseline heat treated samples which were not heated or cooled. Based upon these tests, it is likely that residual thermal stresses cause the slight variations in PS304 coating strength when deposited on different substrates. This effect is eliminated during heat treatment, which allows stress relaxation to occur.

To understand what happens to the coating during the heat treatment metallographic cross sections were made. Figures 9 and 10 show cross-sections of heat treated PS304 on different substrate. The upper sample is the heat-treated coating; the lower is the corresponding sample in the un-heat treated condition. Earlier research on PS304 deposited on 13-8Mo stainless steel, heat treated in air showed the formation of a second phase precipitate in the NiCr matrix. This result is repeated in the current study, figure 9(e). Further, all of the coating samples which were heat treated in air show this same second phase precipitate microstructural feature. Measurements of coating thickness indicate that the heat treatment in air results in a thickness growth of between 5 and 10 percent when compared to the un-heat treated control sample (appearing directly below the heat treated coating sample in each photomicrograph). It is plausible to conclude that coating exposure to high temperature air, which results in the formation of the precipitates in the PS304 binder phase, NiCr, is the cause for the observed cohesive strength and dimensional increases.

In most of the cases (substrate materials) studied no other microstructural changes were observed. In two materials, Inconel 718 and Stellite 6B, the metallographic cross sections reveal the formation of an interfacial reaction zone of layer between the PS304 and the substrate in addition to the formation of the second phase precipitates. For these samples (figs. 11 and 12) the measured thickness changes were comparable to the other cases where no interfacial reaction occurred. This suggests that the interfacial reaction is the result of interdiffusion of species from the coating and the substrate. These types of phenomena are common and may actually enhance the coating to substrate bond strength. This enhancement is the basis of diffusion bonded protective coatings widely employed in the gas turbine industry. Since the PS304 cohesive and adhesive strengths for all of the heat-treated samples exceed the epoxy strength the quantitative effects of the interface diffusion observed in Inconel 718 and Stellite 6B remains speculative.

Figure 13 shows cross sections of Ti-6-4 samples coated with PS304. When heat-treated in argon (fig. 13(a)) the coating microstructure remains unchanged but interfacial delamination, possibly the result of chemical reactions between the titanium and the fluorides, is observed. When heat-treated in air (fig. 13(b)) gross interfacial reactions and complete coating delamination occurred. In addition, the second phase precipitate in the PS304 NiCr matrix is readily seen. This shows that the coating microstructural changes are a result of interactions between the heat treatment atmosphere (air) and the coating and are not a substrate effect.

The metallographic cross-sections of the argon heat-treated samples are shown in figure 10. These photomicrographs show that the PS304 following the heat treatment is virtually indistinguishable from the as-deposited PS304. No second phase precipitates nor interfacial reactions are observed. Measurements also show that no coating thickness changes occur as in the air heat-treated case. Clearly oxygen plays a vital role in the observed properties (adhesion/cohesion) and microstructural changes to heat-treated PS304.

In a previously published report, the authors investigated the second phase precipitates formed during the air heat treatment(s). In that paper, electron microscopy techniques suggested that the precipitates contained high amounts of chromium plus oxygen and traces of silicon. Additional analyses conducted since that work have included x-ray diffraction and x-ray fluorescence microprobe analyses. No conclusive results have been obtained. However, additional research with coating samples containing only NiCr and additional detailed study of PS304 cross-sections offer clues to a mechanism for the formation of the precipitates.

Coating samples of pure, plasma sprayed NiCr were prepared and heat-treated in air or argon at 1200 °F. Adhesion tests were performed and showed that no strengthening effect occurs following the heat treatments in air or argon. Further, cross-sections reveal that no second phase precipitates form even for samples heat-treated in air. Clearly some constituent in PS304 (the Cr_2O_3 , Ag or Fluorides) is playing a role in the formation of the precipitates.

Careful optical observations of PS304 samples heat treated in air (and the corresponding un-heated samples) show that the precipitate formation is closely linked to microstructural proximity of the NiCr phase and the fluoride constituent. In some regions of the PS304 where inhomogeneities exist, little or no precipitates form. For example, NiCr pockets surrounded by Ag or Chrome oxide do not readily segregate

and exhibit the second phase precipitates. Conversely, where the fluorides are in intimate contact with the NiCr (which is virtually everywhere in PS304 since the Fluorides amount to over 20 vol% of the coating) the second phase is always present.

Further research studies are underway to better understand the, perhaps catalytic, role of the fluorides in the formation of the second phase precipitates in the NiCr binder of PS304. In any case, these serendipitous precipitate formations enhance coating strength and seem to create no negative consequences in the performance of PS304. However, in applications where dimensional stability is important, a suitable air heat treatment must be preformed prior to service.

Summary

The effects of substrate and heat treatment atmosphere on the adhesive/cohesive strength of PS304 were studied. It was discovered that, in general, substrate plays a minor role in coating strength when considering as-deposited, un-heat treated coatings. Except for cases where vigorous interfacial reactions were observed (Aluminum and Titanium alloys) substrate has no effect on the strength of heat-treated PS304 coatings.

The results show that heat-treating in argon gas increases strength by approximately 30 percent. This was attributed to interparticle bonded enhanced by atomic diffusion during the heat treatment. No microstructural changes were observed. Heat treating in air resulted in the formation of a second phase precipitate in the NiCr matrix of the PS304 coating. For most of the substrates, no interfacial reactions were observed. The microstructural changes resulted in a thickness increase of about 5 percent suggesting that the precipitates may be due to oxidation. The coating strength increased nearly two-fold following the heat treatment. All of the air heat-treated coatings exhibit the same microstructural features again suggesting that substrate plays no role in coating strength.

The results corroborate earlier work with the PS304 coating and suggest that an air heat treatment be performed to improve coating strength and ensure dimensional stability. Further work is needed to fully understand the mechanism(s) for the observed microstructural and strength changes.

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TABLE I.—COMPOSITION AND SELECTED PROPERTIES OF PS304

Constituent/Property	wt%	Function/Value
NiCr ^a	60	matrix-binder
Cr ₂ O ₃	20	hardener phase
Ag	10	low temperature lubricant
BaF ₂ /CaF ₂ ^b	10	high temperature lubricant
Thermal expansion CTE	--	12.4×10 ⁻⁶ /°C
Density	--	~5.3 g/cc
ρ hardness	--	30 to 34 RC

^aNi/Cr ratio is 80/20 by wt%^bBaF₂/CaF₂ ratio is 62/38 by wt%

TABLE II.—NOMINAL COMPOSITION AND SELECTED PROPERTIES OF COATING SUBSTRATES

Name	Alloy type	Nominal composition, wt%	Thermal conductivity, k W/m-°k	Thermal expansion, CTE ×10 ⁻⁶ /°C
Inconel 718	Nickel base superalloy	53 Ni, 18 Cr, 17 Fe, 5 Nb, 3 Mo, 1 Co, 1 Ti	11.4	14
Stellite 6B	Cobalt base superalloy	54 Co, 29 Cr, 3 Fe, 3 Ni, 5 W, 1.5 Mo, 1 C, 1 Mn	14.9	13.5
304 stainless steel	Austenitic stainless steel	71 Fe, 19 Cr, 10 Ni, 0.1 C	13.8	16
410 stainless steel	Martensitic stainless steel	87 Fe, 13 Cr, 0.2 C	25.1	10
13-8Mo PM	Precipitation hardened stainless steel	76 Fe, 13 Cr, 8 Ni, 2 Mo, 1 Al	15.2	11
Greek Ascolloy	High alloy steel	82 Fe, 13 Cr, 2 Ni, 3 W, 0.2 C	21	10
Ti-6-4	Titanium alloy	90 Ti, 6 Al, 4 V	6.9	10
6061Al	Aluminum alloy	98 Al, 0.6 Si, 0.3 Mn, 1 Mg, 0.2 Cr	167	23

TABLE III.—PULL-OFF COATING STRENGTH DATA SUMMARY

Substrate material	Heat treatment			Comments
	None	Argon	Air HT	
Inconel 718	2890±175 psi	3935±186 psi	4869±354 psi	
	19.9±1.2 MPa	27.1±1.3 MPa	33.6±2.4 MPa	
Stellite 6B	2789±213	3677±136	4945±329	12 samples of each
	19.2±1.5	25.4±0.9	34.1±2.3	
304 stainless steel	2542±362	3627±237	5397±225	
	17.5±2.5	25±1.6	37.2±1.6	
410 stainless steel	2713±62	3229±386	5329±331	
	18.7±0.4	22.3±2.7	36.7±2.3	
13.8 Mo PH	2869±147	3023±269	4605±434	
	19.8±1.0	20.8±1.8	31.8±3.0	
Greek Ascolloy	2619±129	3692±204	5130±235	
	18.1±0.9	25.5±1.4	35.4±1.6	
Ti-6-4	2801±121	--	--	coating delaminated upon heat treatment
	19.3±0.8	--	--	
6061 Al	2366±292	--	--	coating delaminated from heat treatment
	16.3±2.0	--	--	
Average psi	2699±180	3531±337	5046±299	
Average MPa	18.6±1.2	24.3±2.3	34.8±2.1	

TABLE IV.—PULL-OFF DATA SUMMARY OF PREHEATED/PRECOOLED COATED SUBSTRATES

Substrate material	Substrate condition*	Heat treatment	Pull-off strength
6061 Al	Ambient	None	2366±292 psi 16.3±2.0 MPa
6061 Al	Preheated	None	3477±149 23.8±1.0
6061 Al	Precooled	None	1372±225 9.5±1.6
304 stainless steel	Ambient	None	2542±362 17.5±2.5
304 stainless steel	Preheated	None	4178±154 28.8±1.1
304 stainless steel	Precooled	None	2178±549 15.0±3.8
304 stainless steel	Preheated	650 °C/24 hr in air	5639±369 38.8±2.5
304 stainless steel	Precooled	650 °C/24 hr in air	5546±272 38.2±1.9
304 stainless steel	Ambient	650 °C/24 hr in air	5397±225 37.2±1.6

*Ambient means substrate temperature was not controlled, preheat temperature was 260 °C, precooled means 25 °C air jet impinged on sample backside during coating deposition.

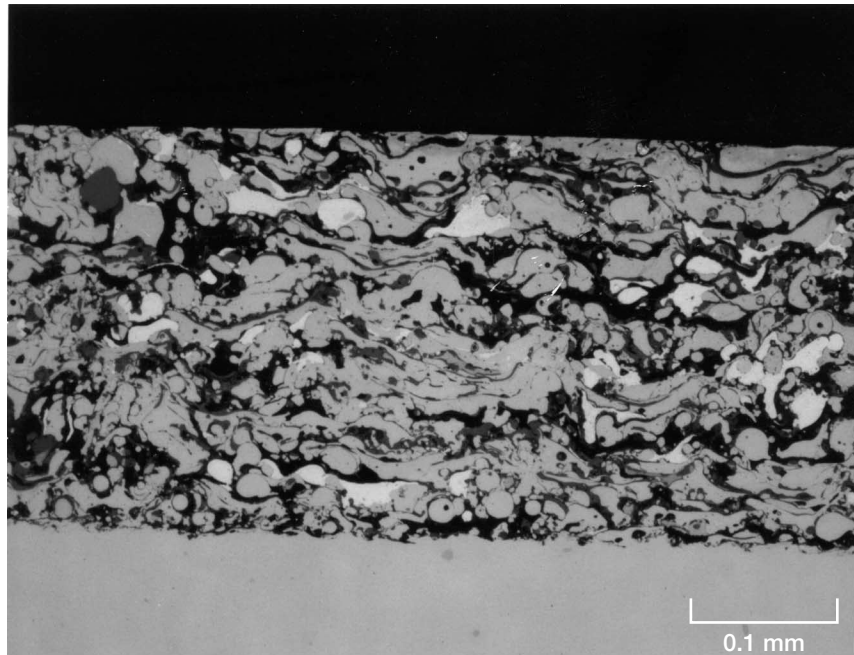


Figure 1.—Typical PS304 cross-section photomicrograph.

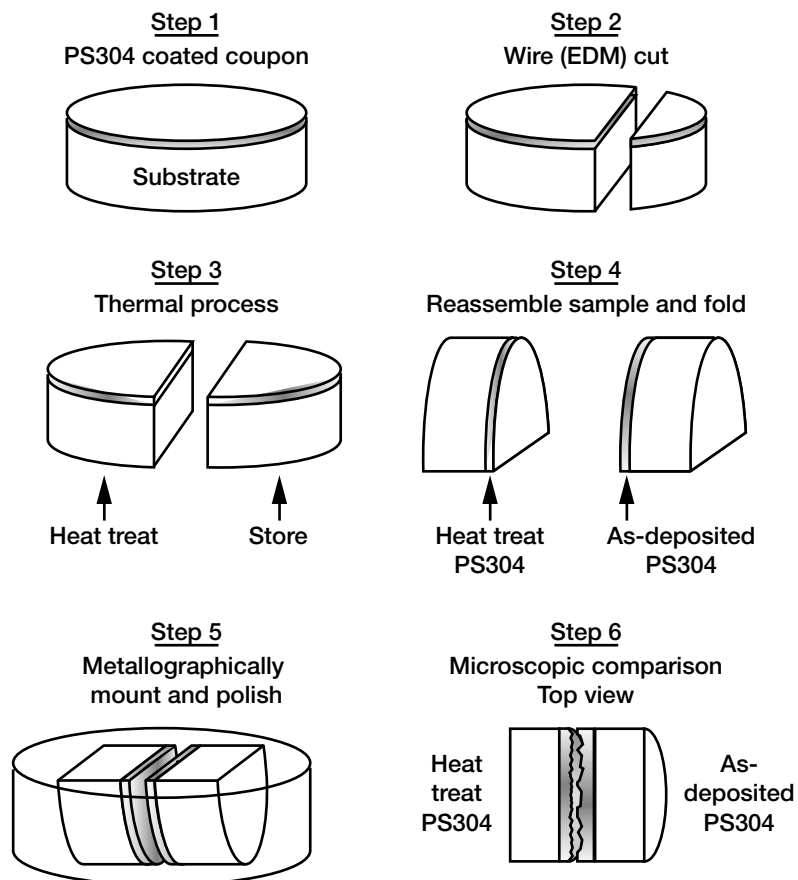
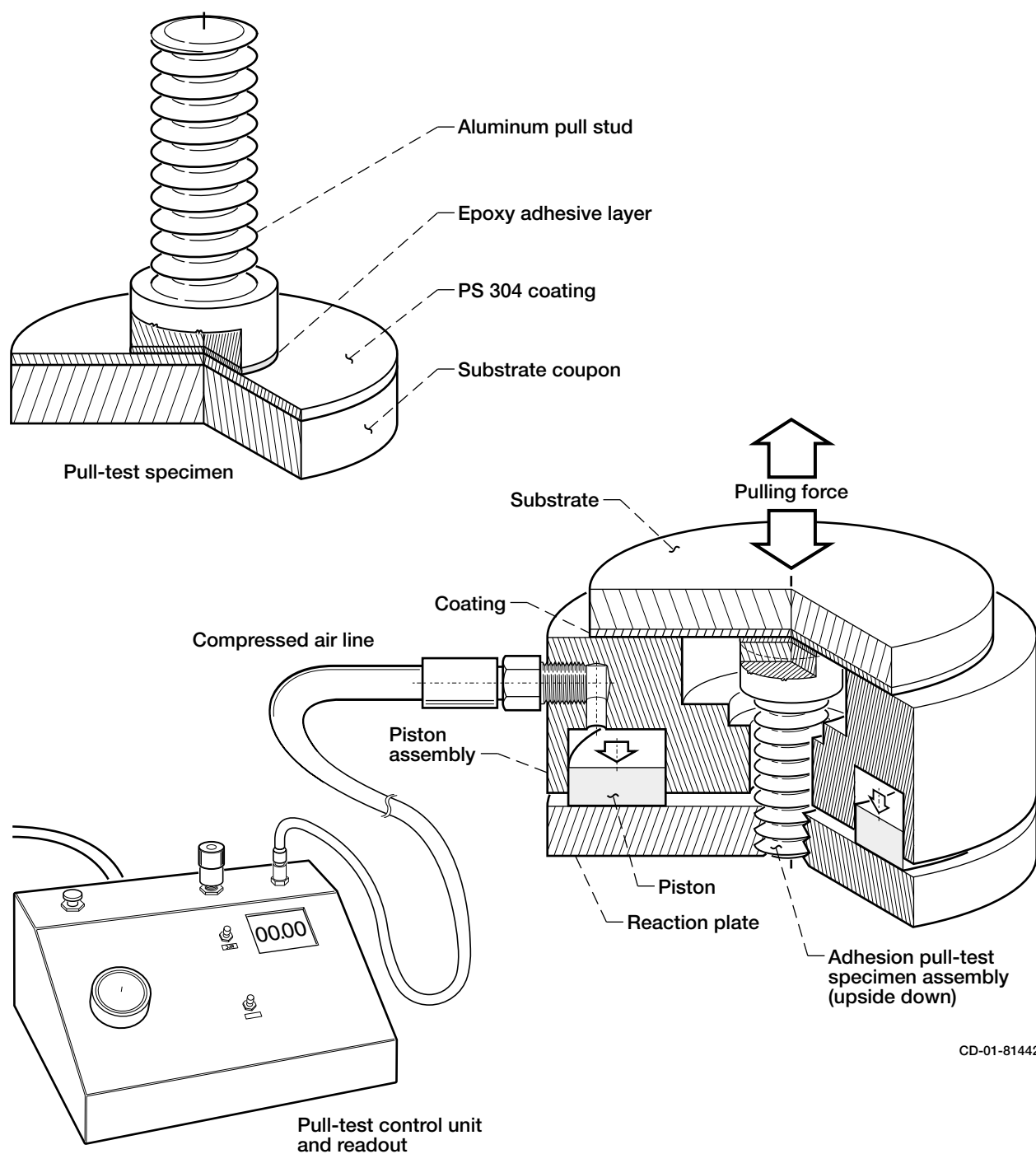


Figure 2.—Schematic representation of cross-sectional mounting technique used to prepare sample.



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Figure 3.—Adhesion pull-test specimen assembly and system.

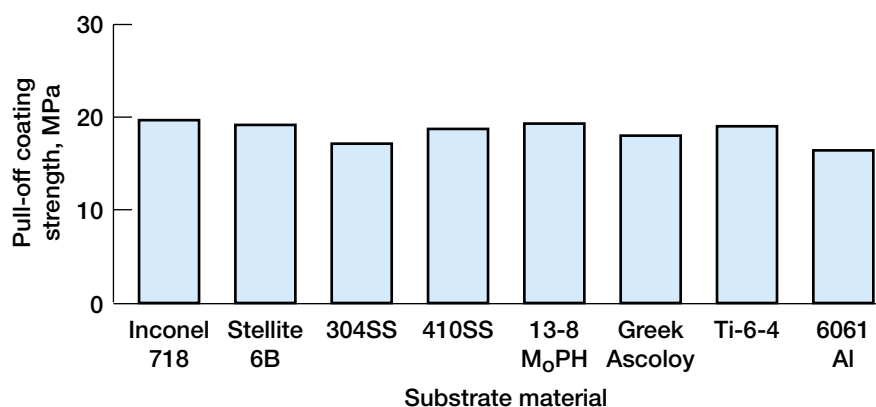


Figure 4.—Coating pull-off strength for the as-deposited (no heat treatment) PS304 on various substrates.

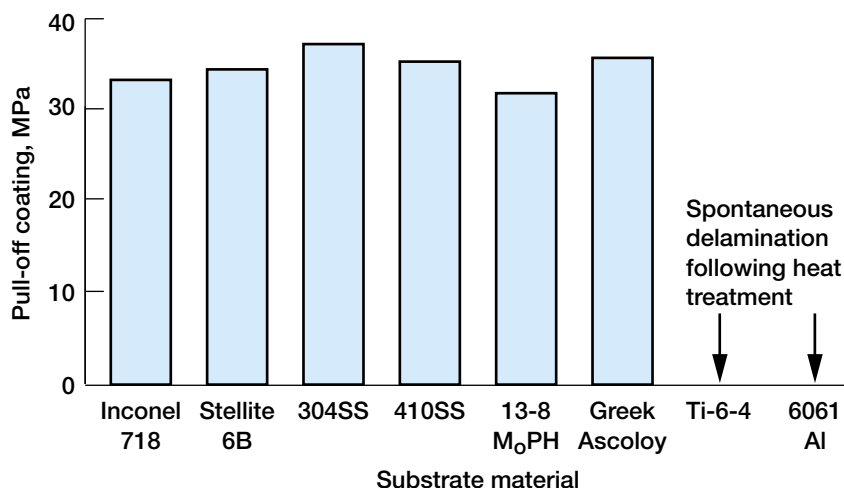


Figure 5.—Coating pull-off strength for the air (100 hr at 650 °C) heat treated PS304 on various substrates. Ti-6-4 and 6061 Al samples delaminated from heat treatment.

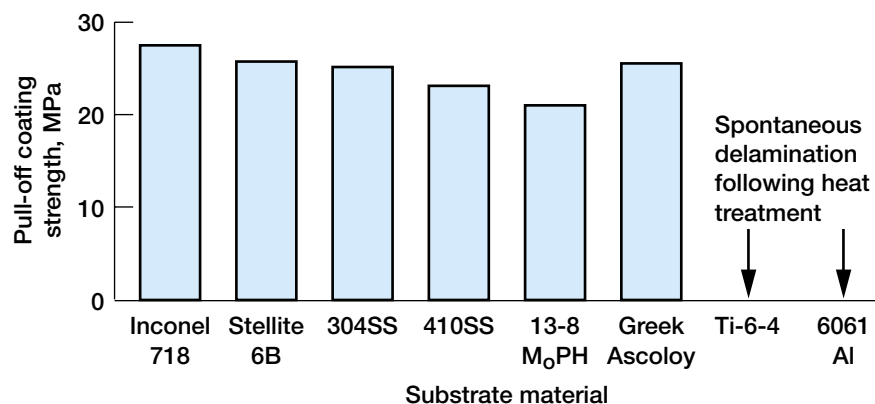


Figure 6.—Coating pull-off strength for the argon (100 hr at 650 °C) heat treated PS304 on various substrates. Ti-6-4 and 6061 Al samples delaminated from heat treatment.

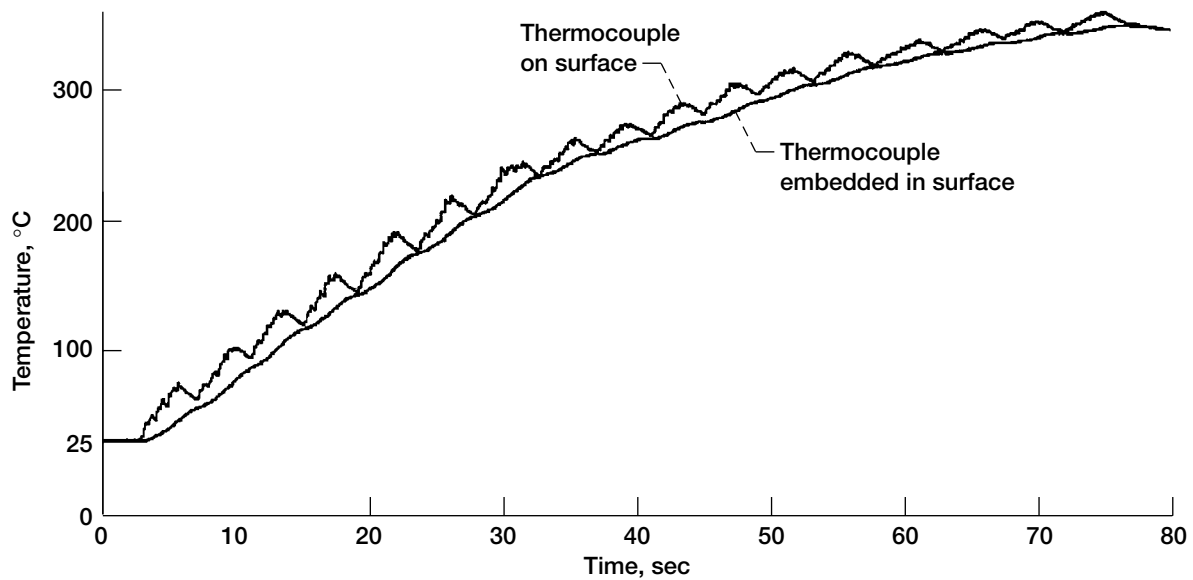


Figure 7.—Substrate temperature during plasma spray deposition PS304 coating on 410 stainless steel.

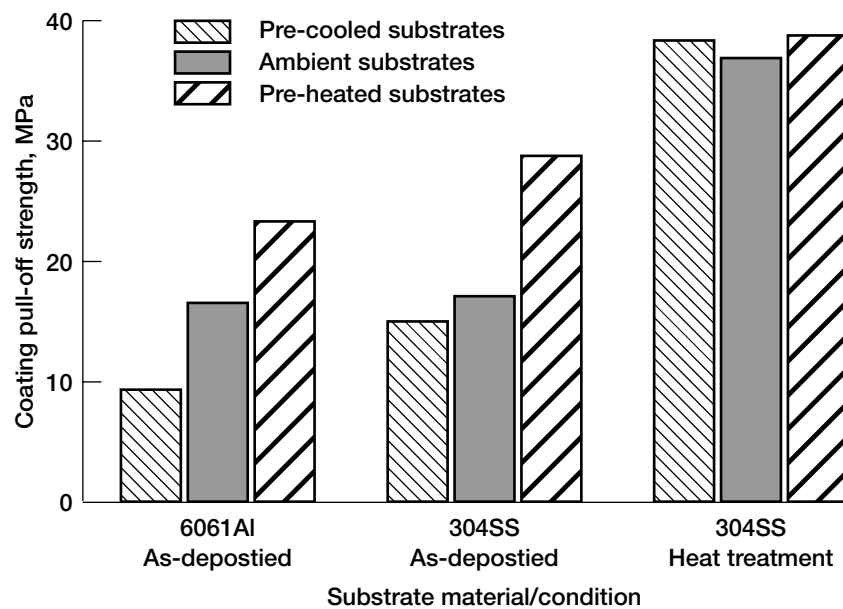


Figure 8.—Coating pull-off strengths for the pre-heated (260 °C) and pre-cooled (25 °C) air impingement) substrates.

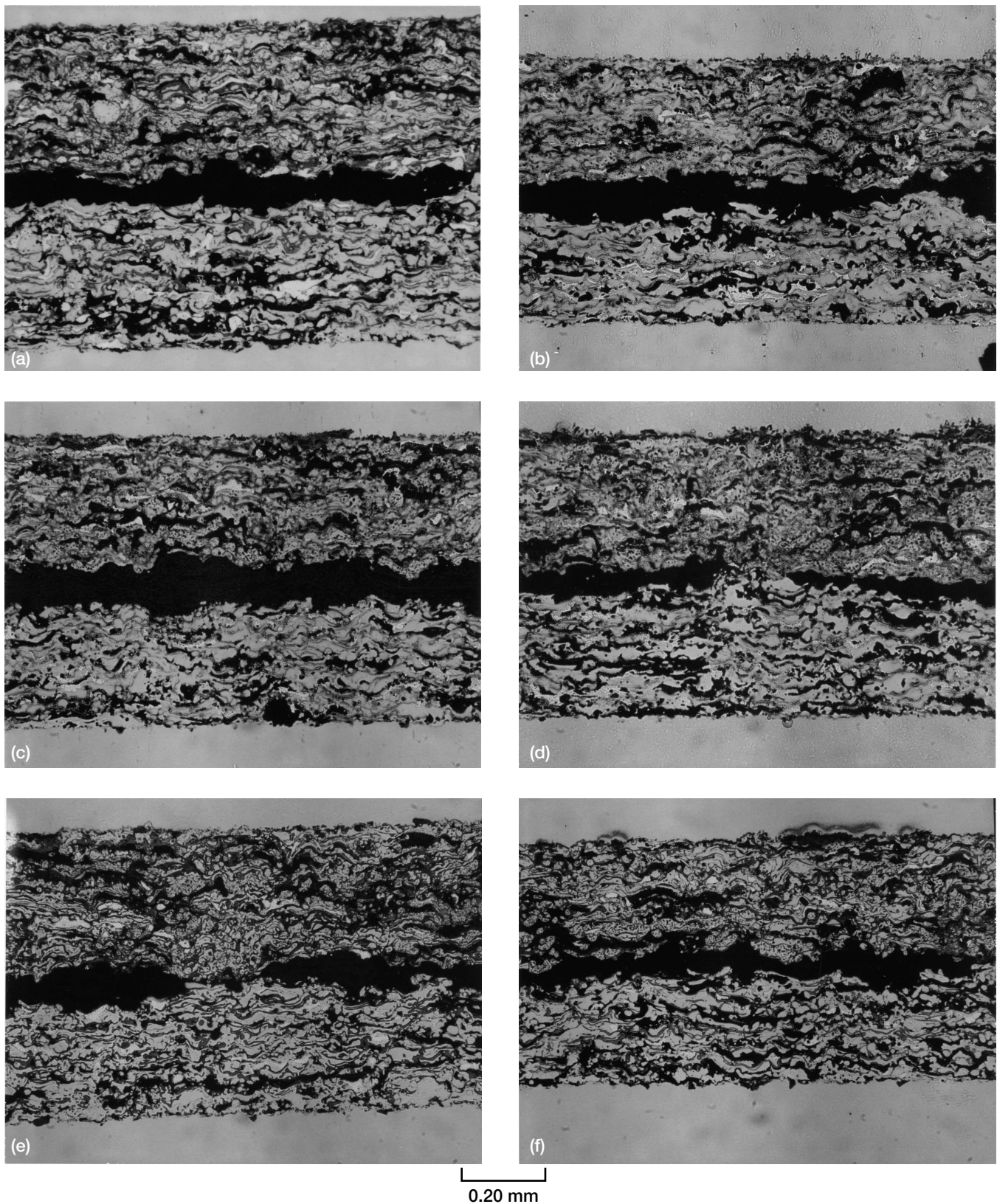


Figure 9.—Metallographic cross-sections of PS304 coatings on various substrates. Upper coating heat treated in air at 650 °C for 100 hr lower coating in as-deposited condition. (a) Inconel 718. (b) Stellite 6B. (c) 304 Stainless steel. (d) 410 Stainless steel. (e) 13-8 MoPH stainless steel. (f) Greek Ascology steel.

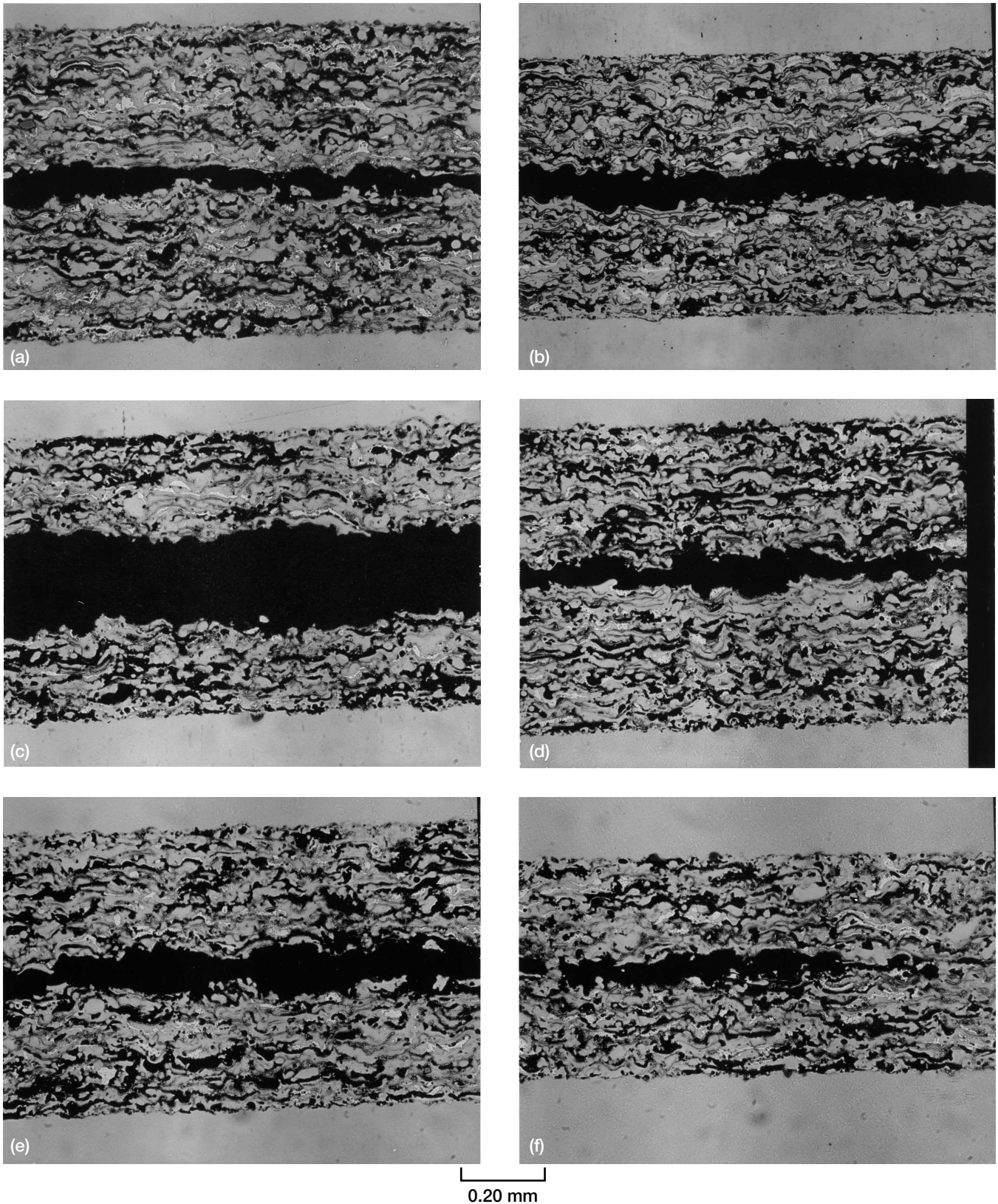


Figure 10.—Metallographic cross-sections of PS304 coatings on various substrates. Upper coating heat treated in argon at 650 °C for 100 hr lower coating in as-deposited condition. (a) Inconel 718. (b) Stellite 6B. (c) 304 Stainless steel. (d) 410 Stainless steel. (e) 13-8 MoPH stainless steel. (f) Greek Ascology high-alloy steel.

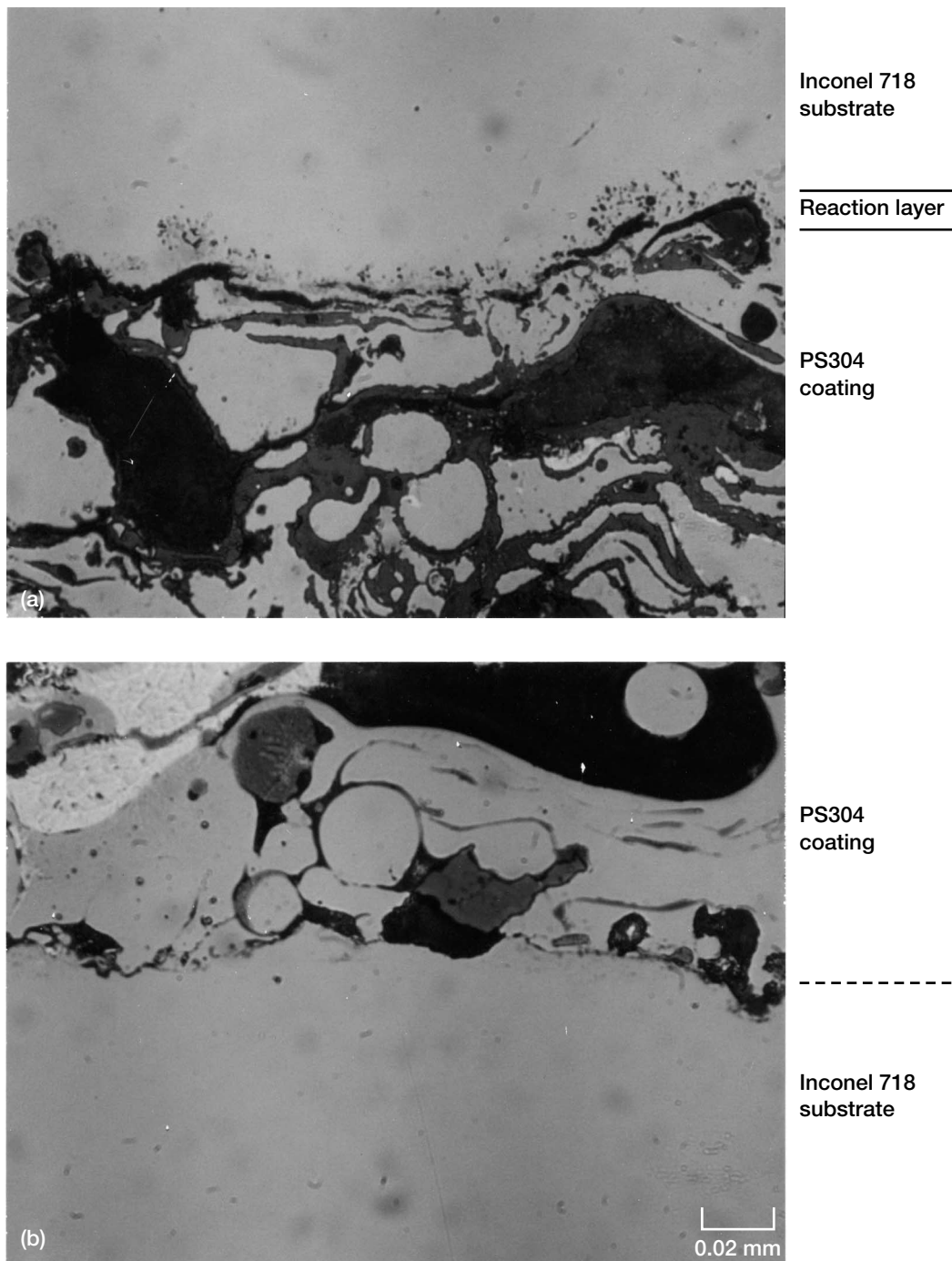


Figure 11.—PS304 coated Inconel 718 sample. (a) Heat treated sample showing reaction layer (dark spots). (b) As deposited (no-heat treatment) shown for comparison.

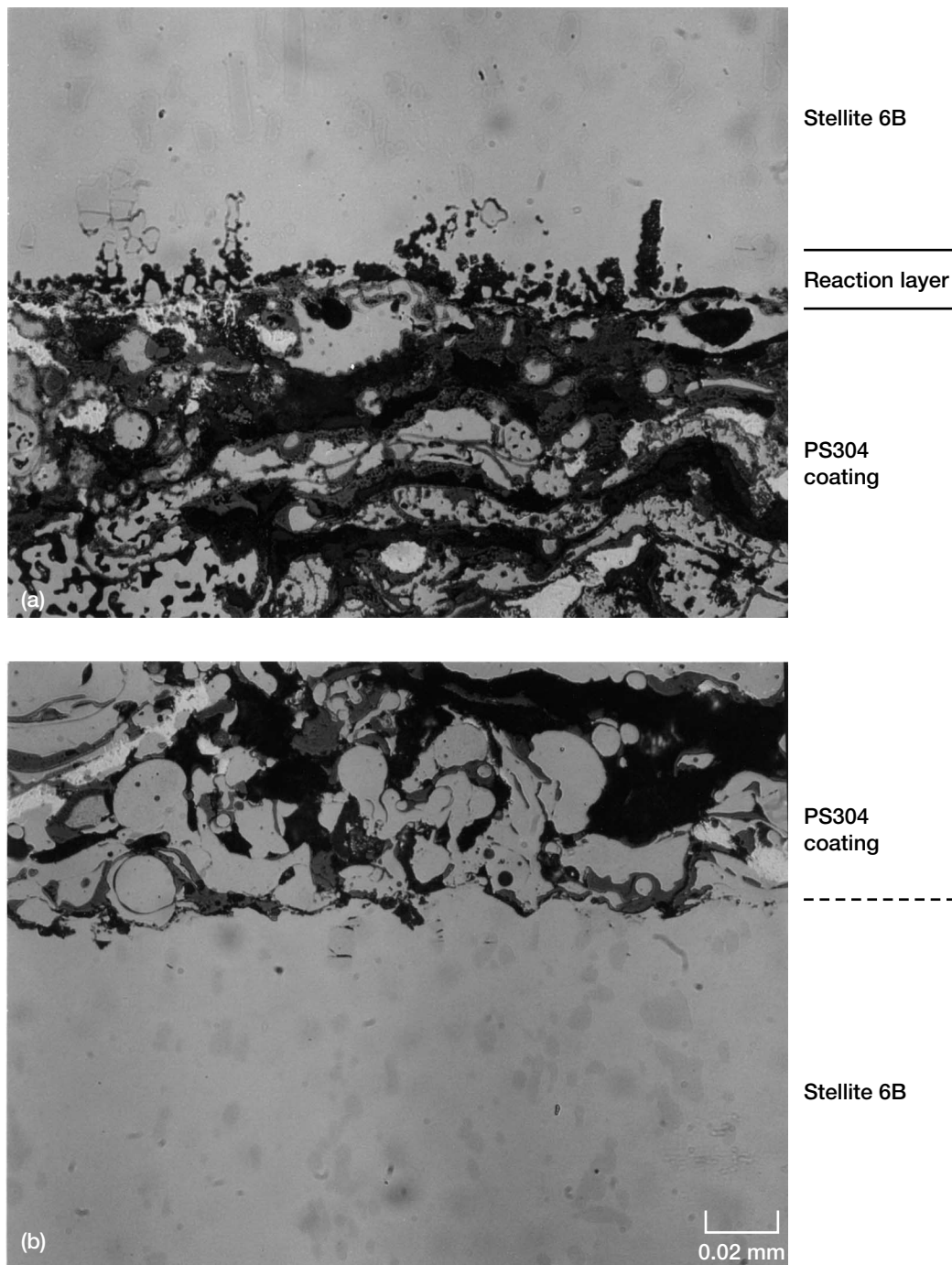


Figure 12.—PS304 coated Stellite 6B sample. (a) Heat treated sample showing interfacial reaction region (dark spots). (b) As deposited (no-heat treatment) shown for comparison.

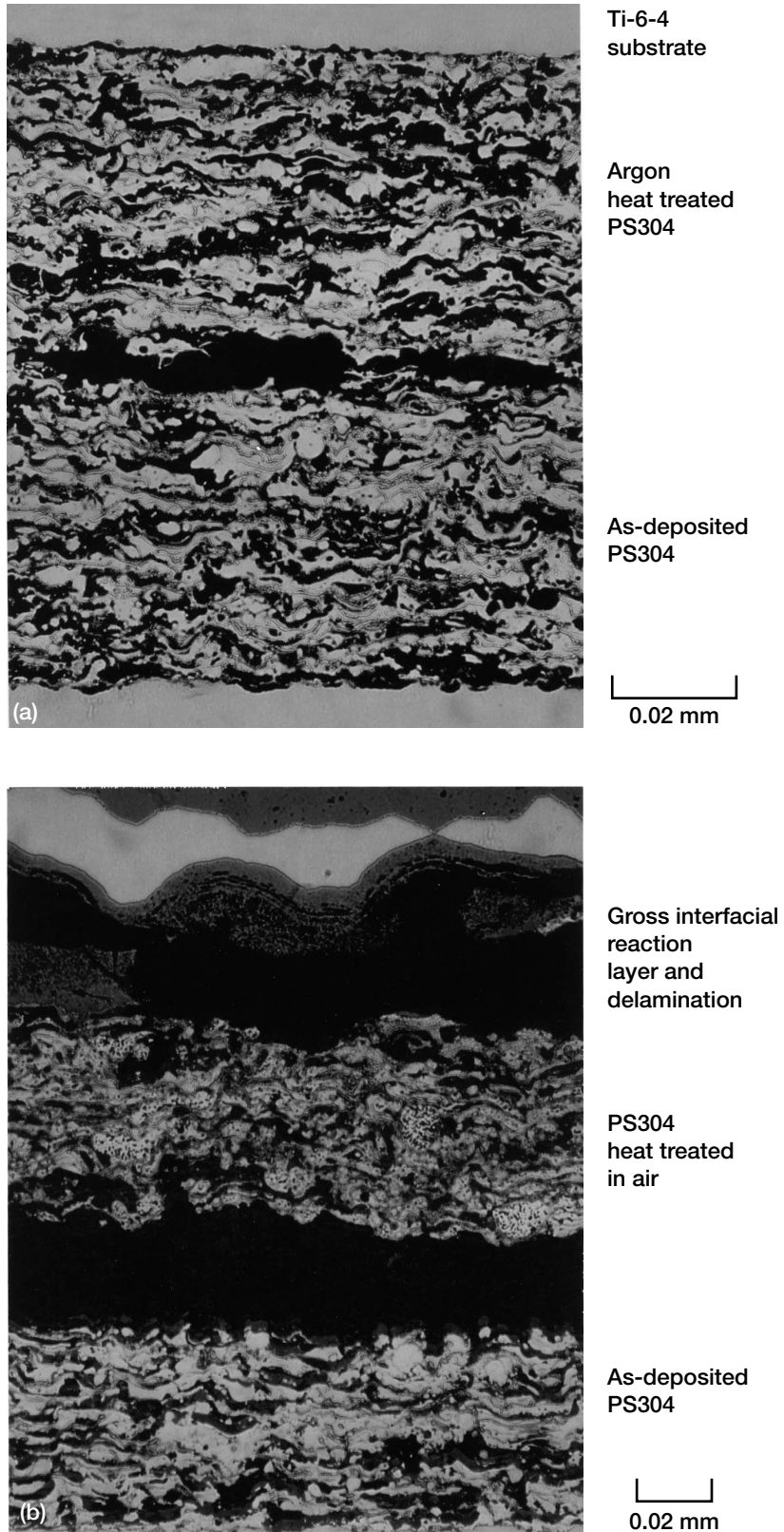


Figure 13.—PS304 coated Ti-6-4 sample. (a) Argon heat treated shows no second phase but delamination at substrate. (b) Air heat treated shows second phase precipitates and gross interfacial reaction layer and delamination.

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13. ABSTRACT (Maximum 200 words) PS304, a plasma spray deposited solid lubricant coating developed for high temperature sliding contacts was deposited on nine different substrate metals, heat treated at 650 °C in either air or argon and subsequently tested for strength using a commercially available pull-off adhesion test. Some samples were examined metallographically to help elucidate and explain the results. As deposited coatings exhibit pull-off strengths typically between 16 and 20 MPa with failure occurring (cohesively) within the coating. Heat treatment in argon at 650 °C results in a slight increase in coating (cohesive) strength of about 30 percent to 21 to 27 MPa. Heat treatment in air at 650 °C results in a dramatic increase in strength to over 30 MPa, exceeding the strength of the epoxy used in the pull test. Cross section metallographic analyses show that no microstructural coating changes occur following the argon heat treatments, however, exposure to air at 650 °C gives rise to the formation of a second chromium-rich phase precipitate within the PS304 NiCr constituent which provides a strengthening effect and a slight (~5 percent) coating thickness increase. Subsequent heat treatments do not result in any further coating changes. Based upon these studies, PS304 is a suitable coating for use on a wide variety of high temperature substrates and must be heat treated following deposition to enhance strength and ensure dimensional stability.				
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