FINAL REPORT
Cooperative Agreement NAG3-2245

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NASA GLENN RESEARCH CENTER

# INVESTIGATION OF POTENTIAL THERMAL PROCESSING TECHNIQUES for the ENHANCEMENT of PS300 HIGH TEMPERATURE SOLID LUBRICANT COATINGS

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## ABSTRACT

Contemporary trends in rotating machinery development have produced a continuous evolution towards ever increasing speeds and higher operating temperatures. This process has been particularly evident in aerospace and automotive applications such as turbochargers. The combination of high temperature and high speed has exceeded the capacity of mainstream liquid lubrication technology. The NASA Glenn Research Center has been at the forefront in developing innovative solid lubricants for the oil free protection of rotating machinery under these extreme environmental conditions. The most recent of these is the PS 300 series of plasma sprayed solid lubricant coatings. St Louis University and NASA Glenn Research Center entered into this cooperative agreement to investigate potential thermal processing techniques for the enhancement of the PS 304 solid lubricant.

## INTRODUCTION

The PS 300 series of plasma sprayed coatings are all $Cr_2O_3$-NiCr based with Ag added for wear reduction at low temperature and a $BaF_2/CaF_2$ eutectic added for protection at high temperature. The coating composition has been optimized by varying the percentages of the above constituents in order to match the thermal expansion properties of the coating to those of the substrate on which it has been sprayed.[1] In particular, the PS 304 coating has proven efficacious in oil free, high speed, high temperature foil bearing applications with superalloy substrates.[2] The feasibility of further enhancement of the PS 304 solid lubricant by post spray thermal processing formed the basis for this cooperative agreement.

Heat treatments are routinely used to modify the structure and/or properties of materials. Hardness, strength, ductility, toughness, adhesion and cohesion[3] are just some of the properties affected by thermal processing. Porosity can also be reduced when plasma sprayed materials are subjected to an appropriate heating, cooling cycle.[4] Treatments such as annealing and tempering can be done before or after final machining. The timing of the heat treatment depends on whether the material modification is used to ease manufacture or enhance the performance of the finished product. In some cases one heat treatment is performed before and a different process is performed after final machining.

The preliminary annealing process for the PS304 coating on superalloy substrates, developed at NASA Glenn, consisted of heating to 1000°F, soaking for 100 hours at temperature, then slow cooling. This process resulted in an increase in coating hardness of 8 - 13%. For obvious economic reasons optimization of this process by reducing the time at temperature would be advantageous. The current study quantified the effect of several different heat treatments on the final hardness of the PS304 coating on three different substrates: Inconel X750, Inconel 718 and 304 stainless steel. As expected the temperature and duration of the anneal process affected the final hardness of the PS304 solid lubricant. The substrate and presence of a bond coat also influenced the pre and post anneal hardness.

## PROCEDURE

NASA Glenn supplied nine sets of test coupons for utilization in this assessment: three each of Inconel X750, Inconel 718 and stainless 304. One set of test coupons of each of the above materials was plasma sprayed with a 0.005 in interlayer bond coat of NiCr and another set of coupons was sprayed with a 0.005 in NiAl bond coat. All of samples were then sprayed with PS304 and then surface ground to leave a 0.015 in PS304 layer. The plasma spray coated substrates were sectioned into smaller samples for heat treatment. All sample preparation was performed at NASA Glenn Research Center.

Coating and substrate hardness were measured and all sample dimensions were recorded prior to thermal processing. A minimum of three Rockwell B, $R_b$, hardness tests were done on each coating and substrate. The coupons were then heated for a proscribed period of time and allowed to cool at room temperature. Hardness and dimensions were then re-measured.

## RESULTS and DISCUSSION

### *Hardness vs. anneal time and temperature*

The first samples were processed utilizing the NASA Glenn baseline procedure of 100 hours at 1000°F. Coating hardness increased between 8 and 13 % with the largest increase in hardness occurring in the NiAl bond coated samples. Tables 1 and 2 contain the raw $R_b$ hardness data and percent increase in hardness respectively. The soaking temperature was then increased by 25% to 1250F while the time at temperature was reduced by 50% to 50 hours. Hardness increases of only a few percent were achieved with this procedure. The annealing temperature was then increased again to 1500 F while the annealing time was held constant at 50 hours. These samples uniformly displayed hardness values less than those of the as received samples. Investigation of these coupons with SEM revealed that substantial degradation of the coatings occurred after being exposed to the 1500 F environment for 50 hours, Fig. 1.

At this point it was decided to substantially reduce the annealing temperature and time and heat the samples incrementally until the hardness of the baseline technique could be reached or exceeded. A set of samples was heated at 1100 F for 25 hours allowed to air cool and then hardness and dimensions were measured. The resulting coating hardnesses were within error measurements of those achieved with the baseline NASA technique. Assuming an Ahrennius type relationship between time, temperature and hardness a simple line graph was extrapolated and an exponential curve fit calculated. Utilizing that plot an annealing temperature of 1170 F for 10 hours was extracted. The results of this heat treatment were for the most part encouraging, 4 - 11% hardness increases overall. Only the samples bond coated with NiAl fell far short of the baseline anneal hardness gains, lagging by up to 75%. A slight increase in temperature to 1200F for the same annealing time, 10 hours, produced $R_b$ values essentially equivalent to the baseline anneal of 100 hours @ 1000 F on the Inconel coupons with a NiCr interfacial layer or no bond layer. All of the stainless 304 coupons and the Inconel coupons with a NiAl interfacial layer still fell short of the baseline anneal levels. A minimal anneal process of 2.5 hrs @ 1200 F resulted in moderate increases in hardness of 3 - 8 %.

*Effect of Substrate and Bond Coat*

The initial coating hardness after plasma spraying but before heat treatment appeared to depend on the substrate and the type of bond coat. Pre-heat coating hardness as a function of substrate and bond coat is shown in Figure 2 with $R_b$ X750 > 718 > 304 regardless of bond coat. All of the annealing processes brought the PS 304 on Inconel 718 hardness up to the same levels as the PS 304 on Inconel X750. The hardness of the plasma sprayed coating on the stainless 304 substrate remained approximately 5% lower overall regardless of the annealing time/temperature. PS 304 hardness on the two Inconels maximized out at about $R_b$ 102 +/- 2. The hardness of PS 304 on stainless 304 never exceeded a $R_b$ of 98.

In addition, the NiAl bond coat appeared to reduce the initial $R_b$ hardness of the PS 304 spray coating by 6 - 8% on all three of the substrates Table 1, Figure 2. Annealing for an appropriate length of time at temperature brought the NiAl bond coated coupons up to about the same $R_b$ hardness levels as the other coatings on the same substrates, Table 1 (1000 F/100 hrs, 1100 F/25 hrs). Annealing at a higher temperature for a shorter time (1200F/10 hrs) developed hardness levels roughly equivalent to those of the baseline procedure on coupons with an NiCr bond coat or no bond layer but not on those with the NiAl bond layer.

Hardness vs. anneal process plots for all of the PS 304/bondcoat/substrate combinations and for the substrates themselves are included in appendix A.

*Thermal expansion*

An unsuccessful attempt was made to correlate the linear and volumetric thermal expansion rates with the anneal process. This was not possible with the available samples because the variability of the coupon dimensions was of the same order as the apparent post process change in dimension. This data has not been included in this report but is available upon request.

## Conclusions and Project Achievements

Substrate and interfacial bond layer affect both the as sprayed and the post anneal hardness of the PS 304 solid lubricant coating. Therefore, a single one size fits all anneal process may not be achievable or desirable. This study produced several more efficient thermal enhancement processes for the PS 304 solid lubricant coatings. An increase in anneal temperature from 1000°C to 1100°C coupled with a decrease in anneal time from 100 hours to 25 hours to produced an equivalent increase in coating hardness on all substrate/bond coat combinations tested. Substrate/bond coat specific anneal techniques appeared be necessary when further anneal time minimization was attempted. A time/temperature combination of 10 hrs @ 1200°F produced satisfactory results on both of the Inconels with no interfacial layer as well as with a NiCr bond coat. However, this process produced unsatisfactory results on the stainless 304 with all bond coat combinations and on the Inconels with the NiAl bond coat. A longer soak time appeared to be necessary to achieve similar coating enhancements with these substrate/bond coat combinations.

A more in depth investigation of the substrate/bond coat affect on PS 304 hardness and other properties is needed. An extension of this co-operative agreement to further study the limits of the PS 304 plasma sprayed lubricant is under development.

An abstract delineating the results of this co-operative agreement has been submitted for presentation at the 2000 ASM International Materials Solutions Conference in St. Louis, Mo. October, 2000.

TABLE 1: Rockwell B hardness vs. anneal method

| SUBSTRATES | | Inconel x750 | | | Inconel 718 | | | 304 Stainless | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Bond Coat | | None | NiAl | NiCr | None | NiAl | NiCr | None | NiAl | NiCr |
| Anneal Temp (F) | Anneal Time (hrs) | | | | | | | | | |
| - | - | 96.0 ± 1.6 | 89.3 ± 1.4 | 95.0 ± 1.6 | 93.3 ± 0.9 | 87.5 ± 2.2 | 90.5 ± 1.8 | 90.2 ± 1.2 | 85.0 ± 0.6 | 88.2 ± 1.2 |
| 1000 | 100 | 104.4 ± 0.4 | 100.0 ± 0.1 | 103.1 ± 0.5 | 102.7 ± 1.0 | 99.1 ± 1.4 | 100.9 ± 0.3 | 97.7 ± 0.9 | 95.9 ± 0.7 | 96.2 ± 0.4 |
| 1250 | 50 | 97.1 ± 1.1 | 89.9 ± 0.7 | 96.4 ±0.7 | 100.1 ± 1.0 | 90.0 ± 0.5 | 95.3 ± 0.3 | 91.5 ± 0.6 | 84.1 ± 0.9 | 92.5 ± 1.9 |
| 1500 | 50 | 90.7 ± 1.2 | 83.6 ± 0.5 | 84.0 ± 1.3 | 97.0 ± 1.3 | 78.6 ± 2.6 | 79.1 ± 2.5 | 81.5 ± 0.5 | 76.5 ± 1.4 | 79.4 ± 0.8 |
| 1100 | 25 | 102.0 ± 1.7 | 100.4 ± 0.4 | 99.9 ± 1.9 | 102.1 ± 1.1 | 100.4 ± 0.3 | 101.0 ± 1.0 | 96.5 ± 0.9 | 95.7 ± 0.3 | 96.2 ± 0.4 |
| 1170 | 10 | 101.0 ± 1.3 | 92.7 ± 0.8 | 99.8 ± 0.5 | 102.5 ± 0.4 | 97.0 ± 0.5 | 98.6 ± 0.8 | 94.8 ± 0.8 | 90.0 ± 1.3 | 94.9 ± 1.3 |
| 1200 | 10 | 102.9 ± 1.2 | 96.6 ± 0.6 | 103.2 ± 0.9 | 102.0 ± 0.1 | 93.0 ± 0.5 | 98.9 ± 0.9 | 95.1 ± 0.4 | 92.1 ± 0.9 | 93.1 ± 0.9 |
| 1200 | 2.5 | 100.7 ± 1.0 | 95.4 ± 0.7 | 101.2 ±0.3 | 99.5 ± 0.3 | 94.9 ± 1.3 | 97.9 ± 1.1 | 93.4 ± 0.7 | 90.7 ± 1.3 | 93.2 ± 1.6 |

TABLE 2: % increase in Rockwell B hardness vs. anneal method

| SUBSTRATES | | Inconel x750 | | | Inconel 718 | | | 304 Stainless | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Bond Coat | | None | NiAl | NiCr | None | NiAl | NiCr | None | NiAl | NiCr |
| Anneal Temp (F) | Anneal Time (hrs) | | | | | | | | | |
| - | - | 96.0 ± 1.6 | 89.3 ± 1.4 | 95.0 ± 1.6 | 93.3 ± 0.9 | 87.5 ± 2.2 | 90.5 ± 1.8 | 90.2 ± 1.2 | 85.0 ± 0.6 | 88.2 ± 1.2 |
| 1000 | 100 | 8.8 | 12.0 | 8.5 | 10.1 | 13.6 | 11.5 | 8.3 | 12.8 | 9.1 |
| 1250 | 50 | 1.1 | 0.7 | 1.5 | 7.3 | 3.2 | 5.3 | 1.4 | - 1.1 | 4.9 |
| 1500 | 50 | - 5.5 | -6.4 | - 11.5 | 4.0 | - 9.8 | - 12.6 | - 9.6 | - 10. | - 10 |
| 1100 | 25 | 6.3 | 12.4 | 5.2 | 9.4 | 15.1 | 11.6 | 7.0 | 12.6 | 9.1 |
| 1170 | 10 | 5.2 | 3.8 | 5.1 | 9.9 | 11.2 | 9.0 | 5.1 | 5.9 | 7.6 |
| 1200 | 10 | 7.8 | 8.2 | 8.6 | 9.3 | 6.6 | 9.3 | 5.4 | 8.4 | 5.6 |
| 1200 | 2.5 | 4.9 | 6.8 | 6.5 | 6.6 | 8.8 | 8.2 | 3.5 | 6.7 | 5.7 |

Figure 2: Preheat coating hardness vs. substrate/bond coat

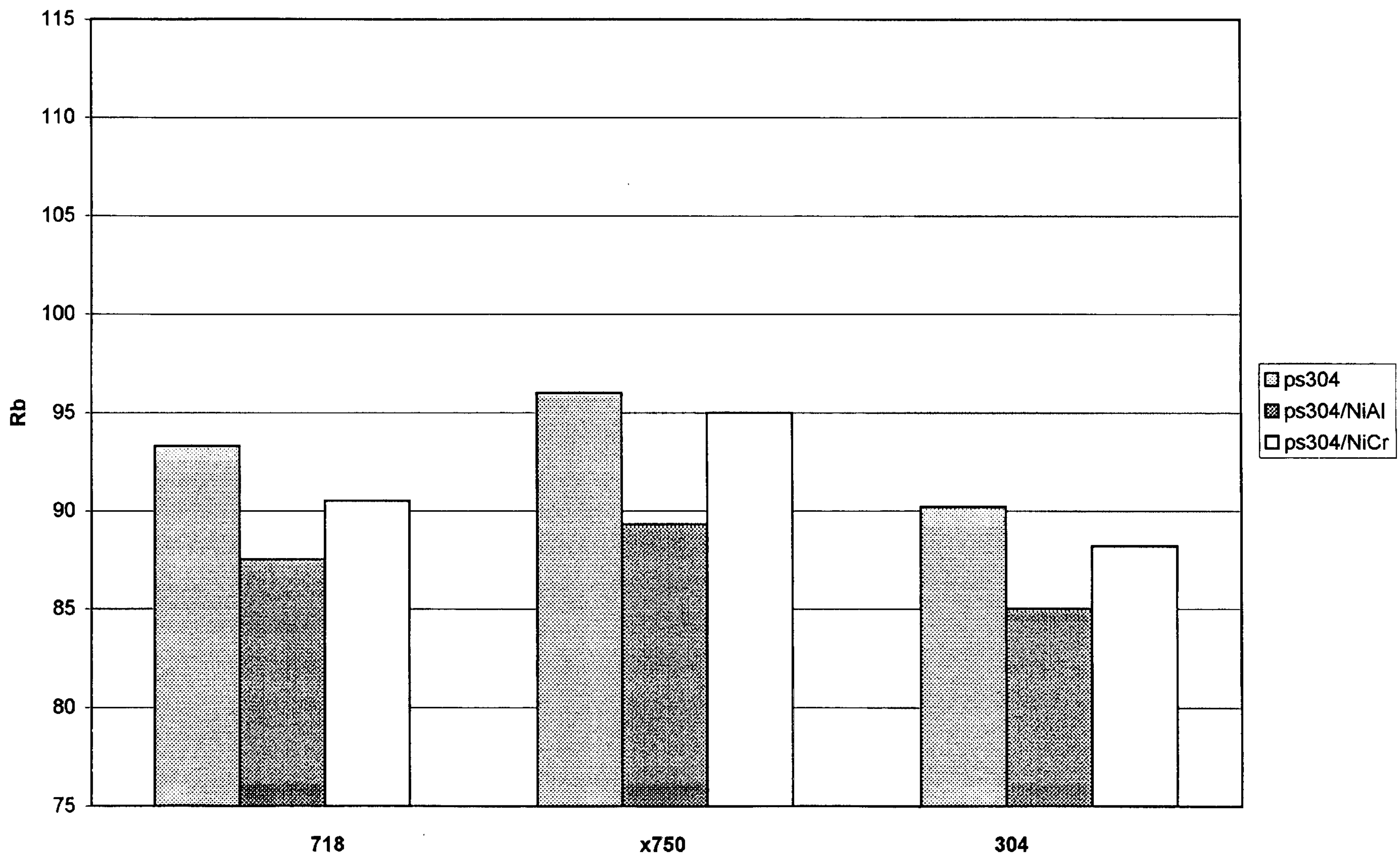
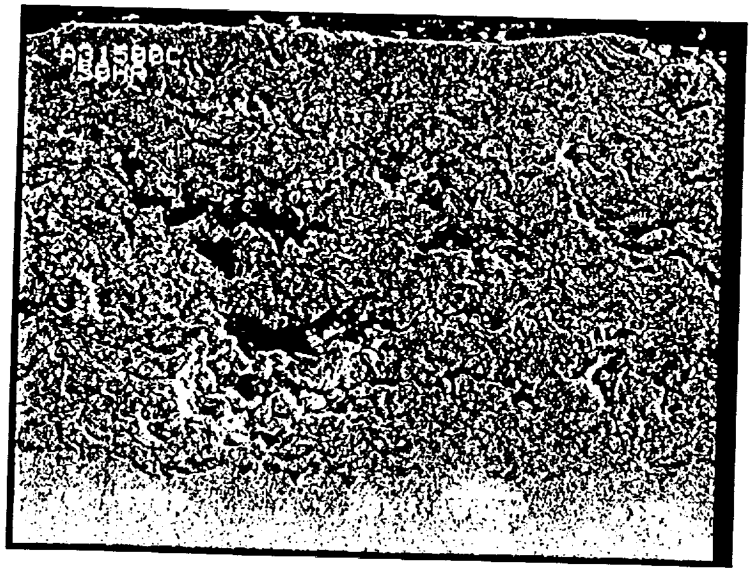
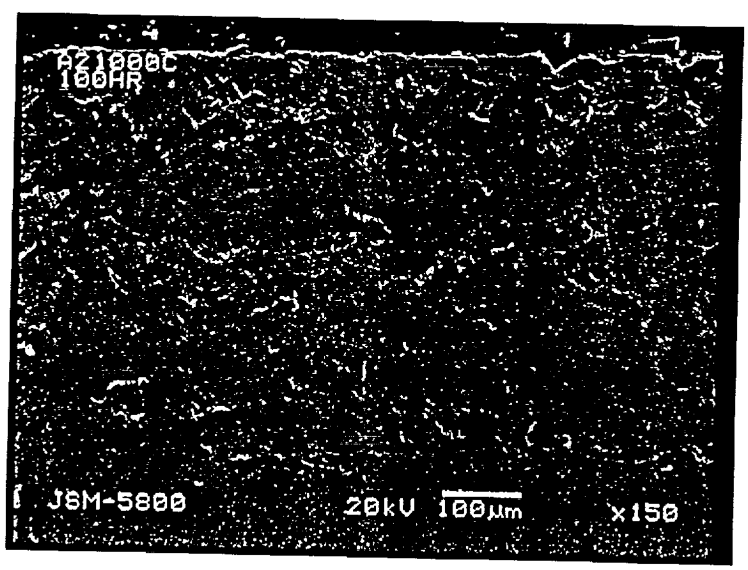


Figure 2: PS 304 coating after 1500 F for 50 hrs. Note voids and cracks. The same coating after 1000 C for 100 hrs is included for comparison.



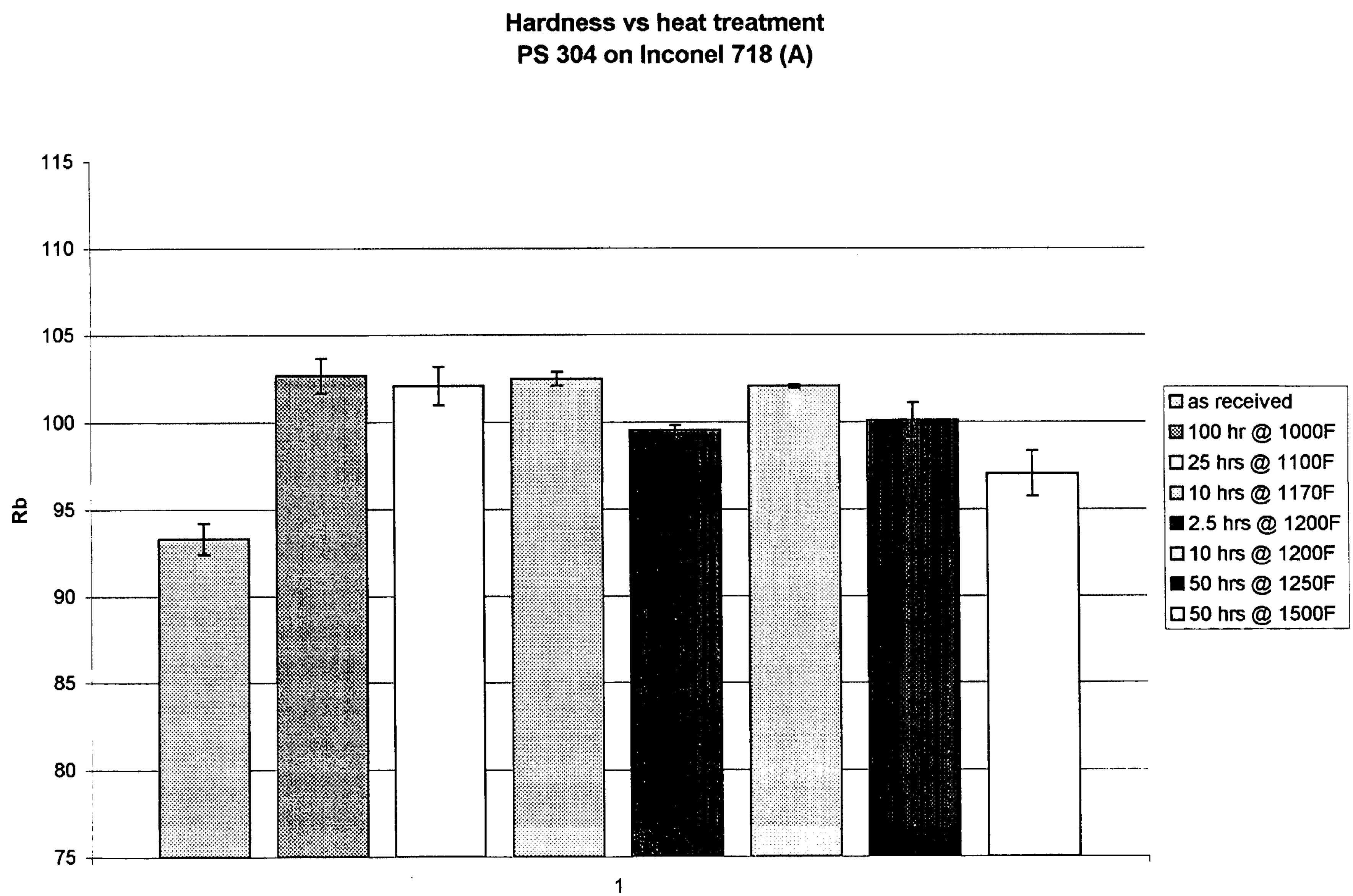
[1] DellaCorte, C., Fellenstein, J. A., "The Effect of Compositional Tailoring on the Thermal Expansion and Tribological Properties of PS300: A Solid Lubricant Composite," NASA TM 834 49, 10/96.

[2] DellaCorte, C., Fellenstein, J. A., and Benoy, P. A., "Evaluation of Advanced Solid Lubricant Coatings for Foil Air Bearings Operating at 25 and 500°C," presented at the STLE/ASME Joint Tribology Conference 10/98 in Toronto, Canada and accepted for publication in Tribology Transactions.

[3] Hennaut, J., Othmezouri, J., and Charlier, J., "Influence of Heat Treatments on the Cohesion and Adhesion Strength of Plasma-Sprayed Deposits", Thin Solid Films, 192, pp. 97-109, 1990.

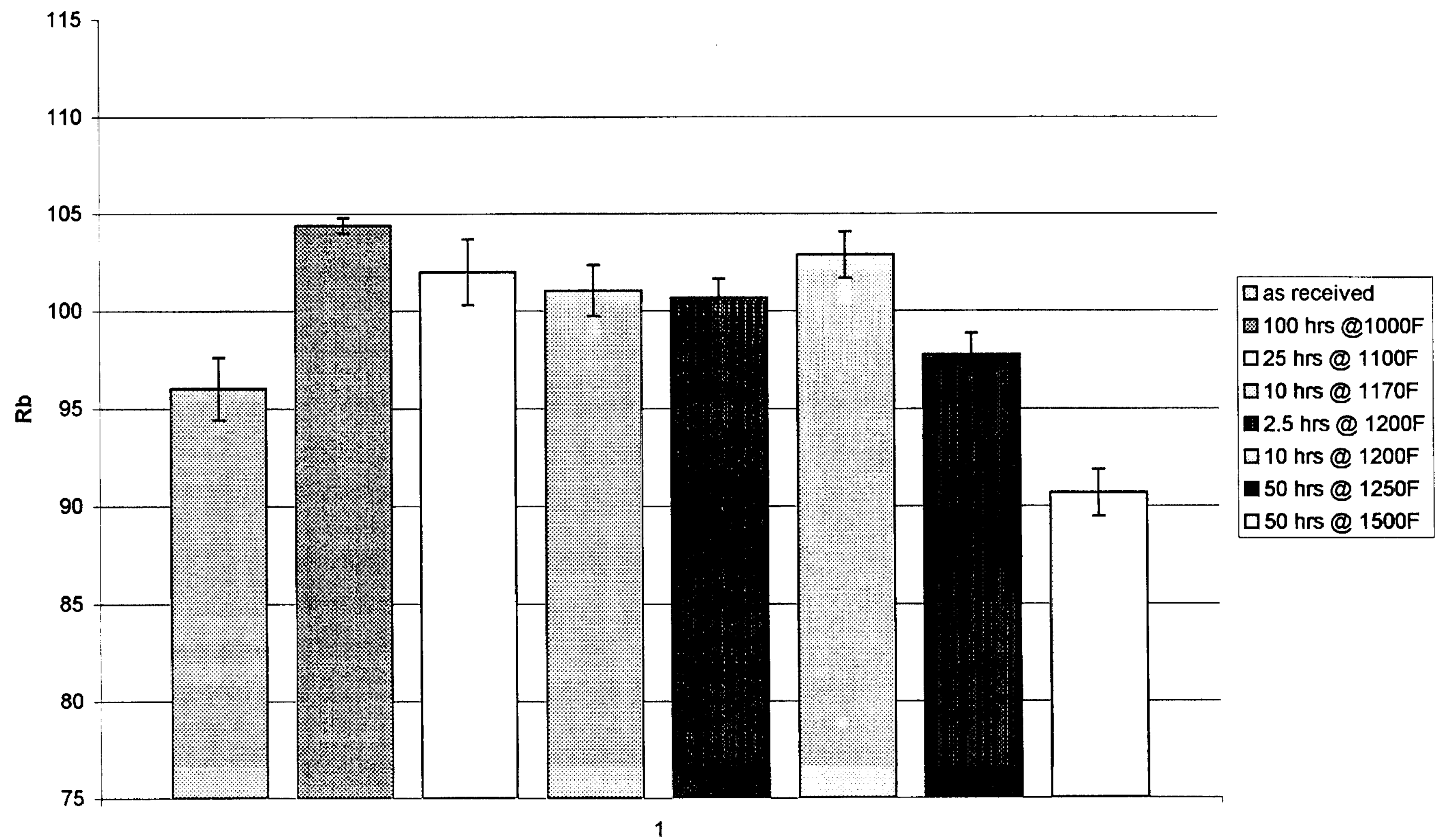
[4] Kim, D.-Y., Han, M.-S., and Youn, J.-G., "Characterization of Erosion Resistant $Cr_3C_2$-NiCr Plasma Sprayed Coatings," Thermal Spray: Practical Solutions for Engineering Problems, ASM International, USA, pp. 123-128, 1996.

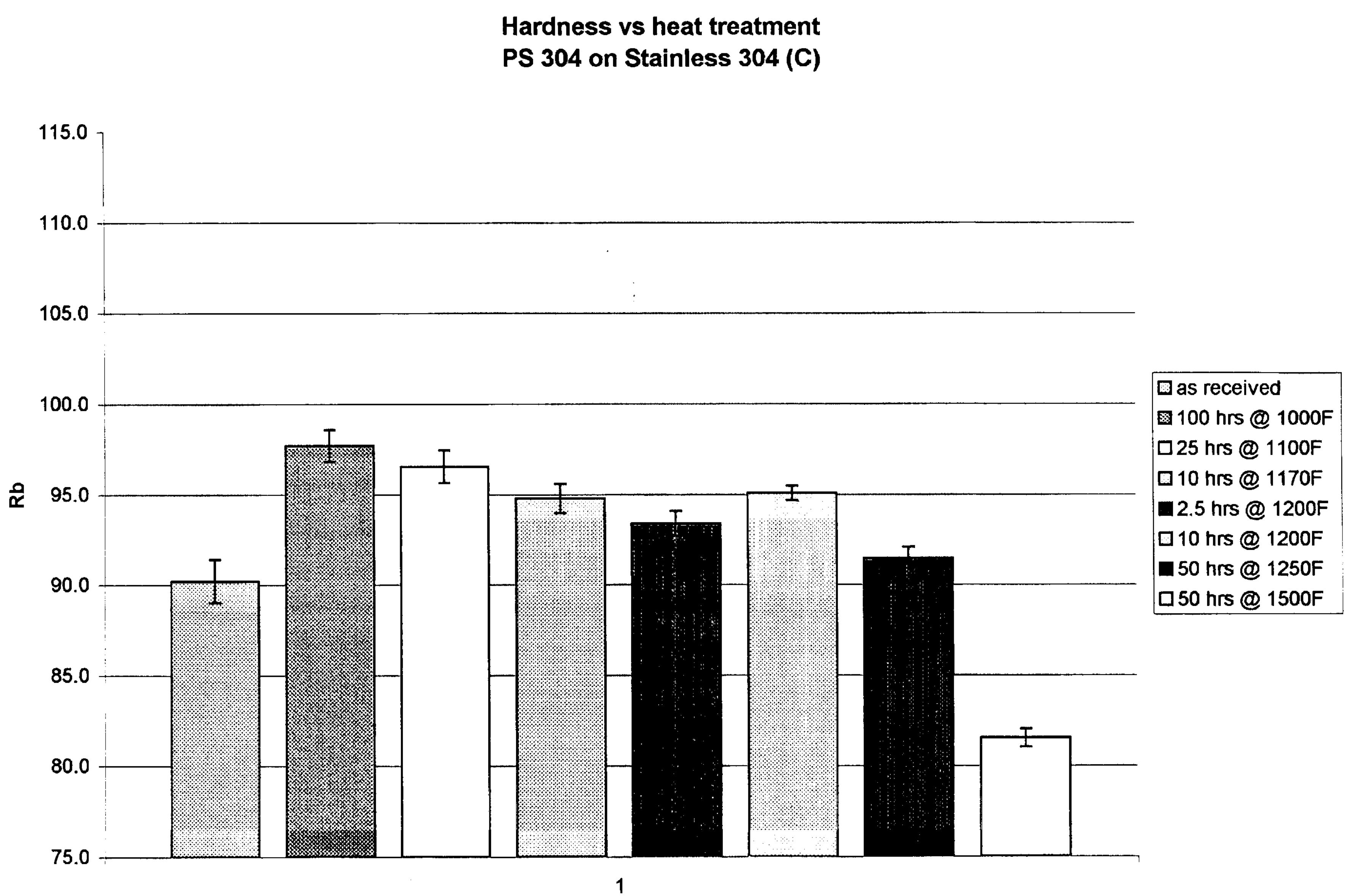
# APPENDIX A
# ROCKWELL B HARDNESS vs. HEAT TREATMENT
# ALL SUBSTRATES and COATING COMBINATIONS

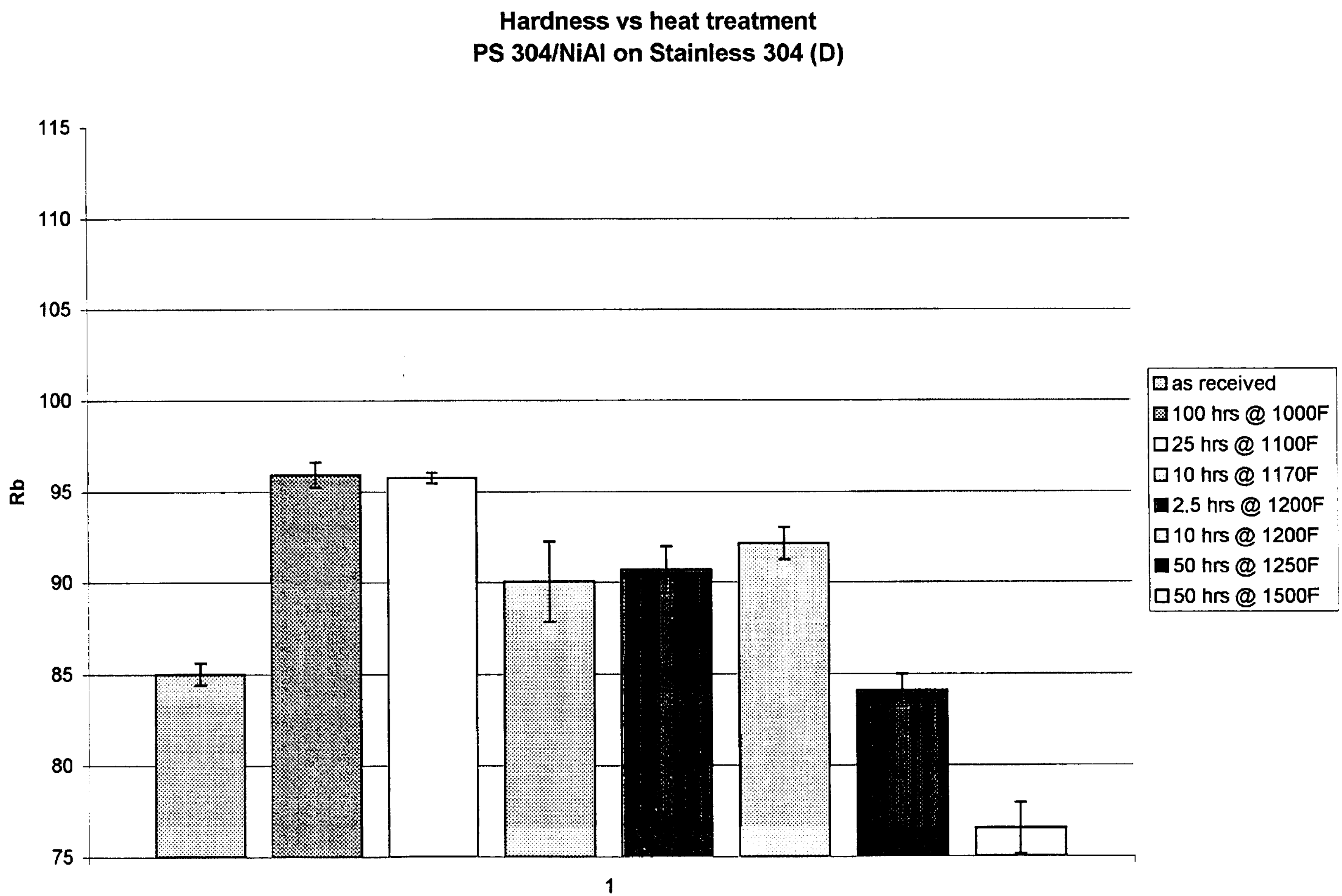


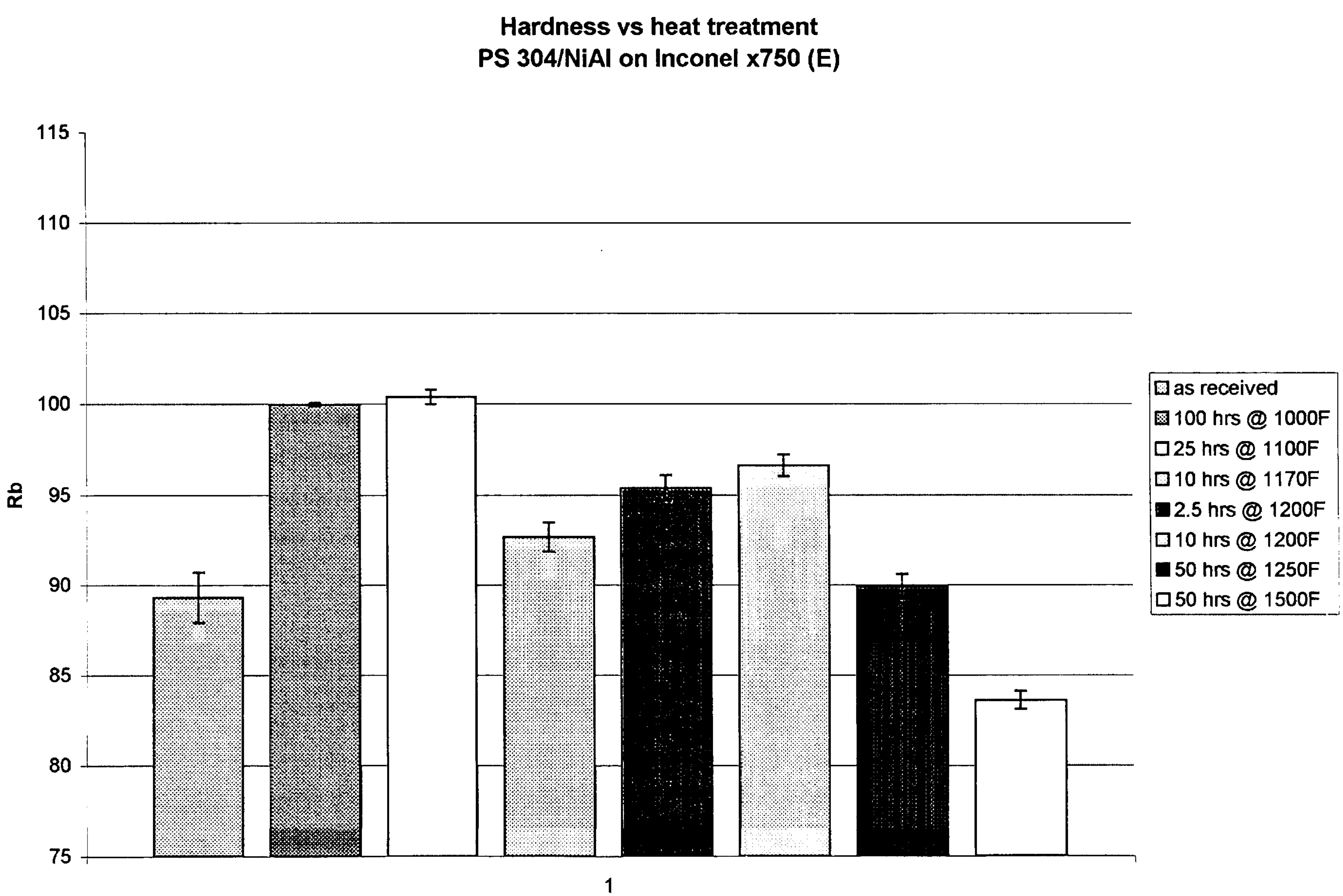
Hardness vs heat treatment
PS 304 on Inconel 718 (A)
Rb
115
110
105
100
95
90
85
80
75
1
as received
100 hr @ 1000F
25 hrs @ 1100F
10 hrs @ 1170F
2.5 hrs @ 1200F
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500F

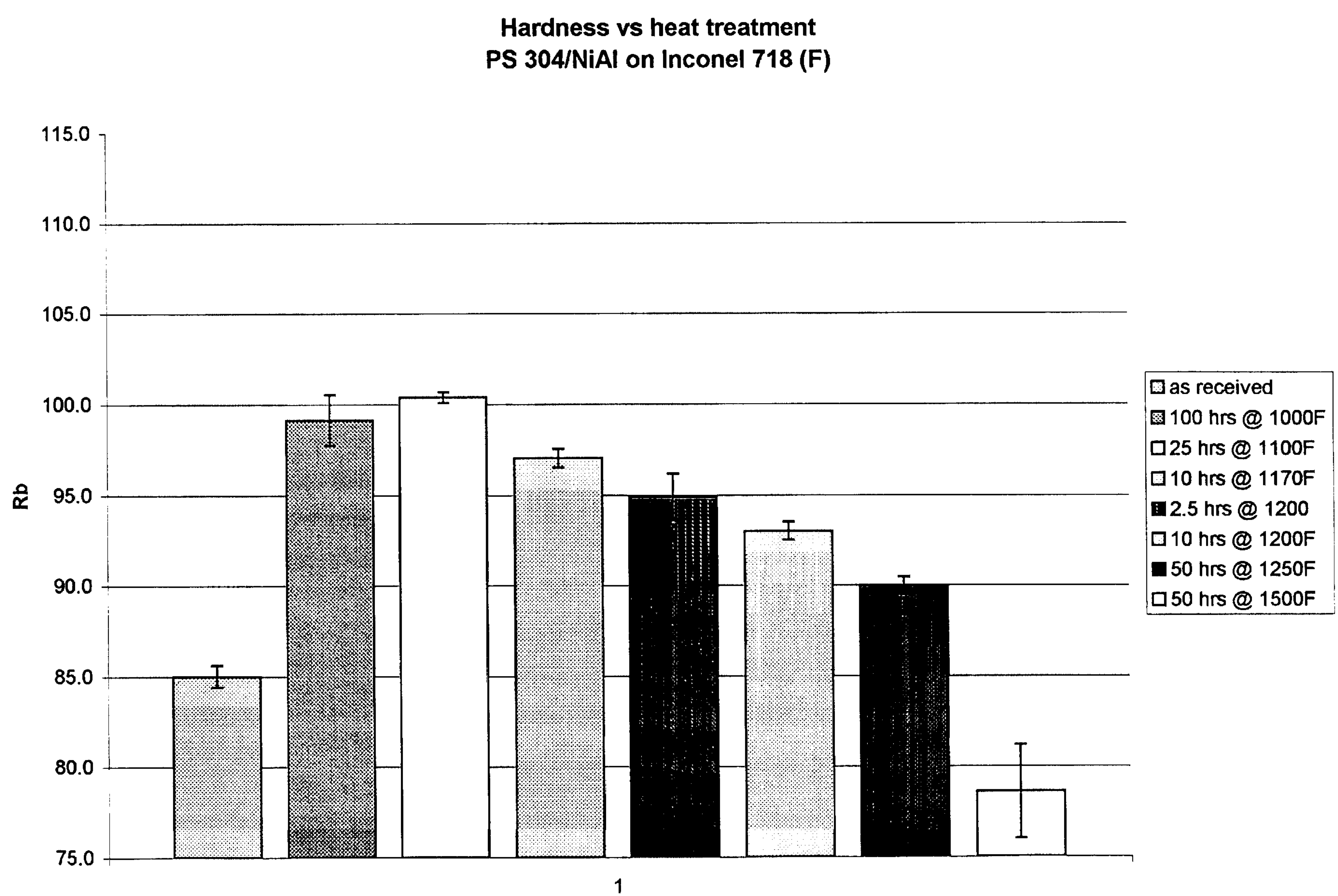
## Hardness vs heat treatment
## PS 304 on Inconel x750 (B)



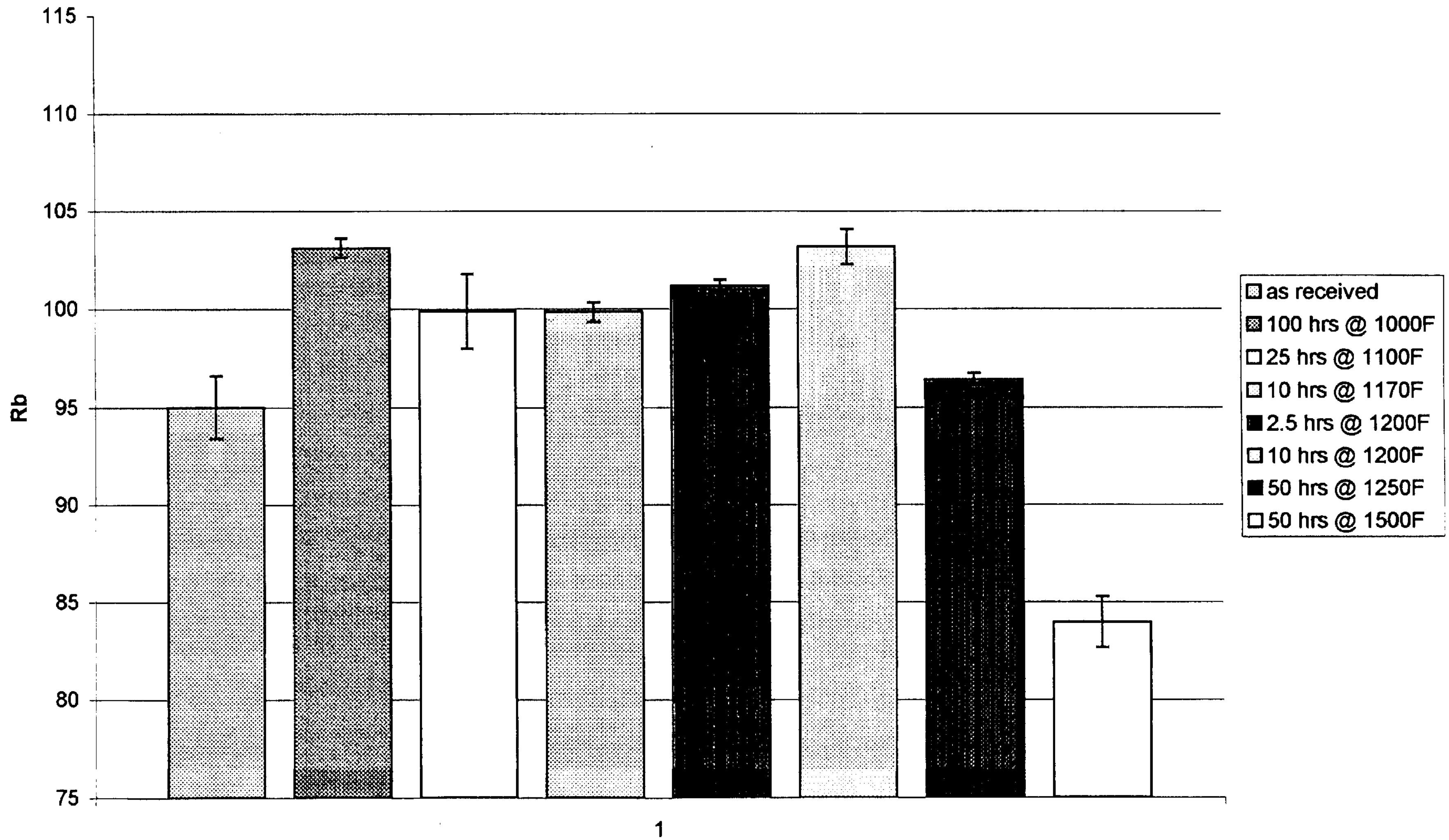

Hardness vs heat treatment
PS 304 on Stainless 304 (C)
115.0
110.0
105.0
100.0
95.0
90.0
85.0
80.0
75.0
Rb
1
as received
100 hrs @ 1000F
25 hrs @ 1100F
10 hrs @ 1170F
2.5 hrs @ 1200F
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500F

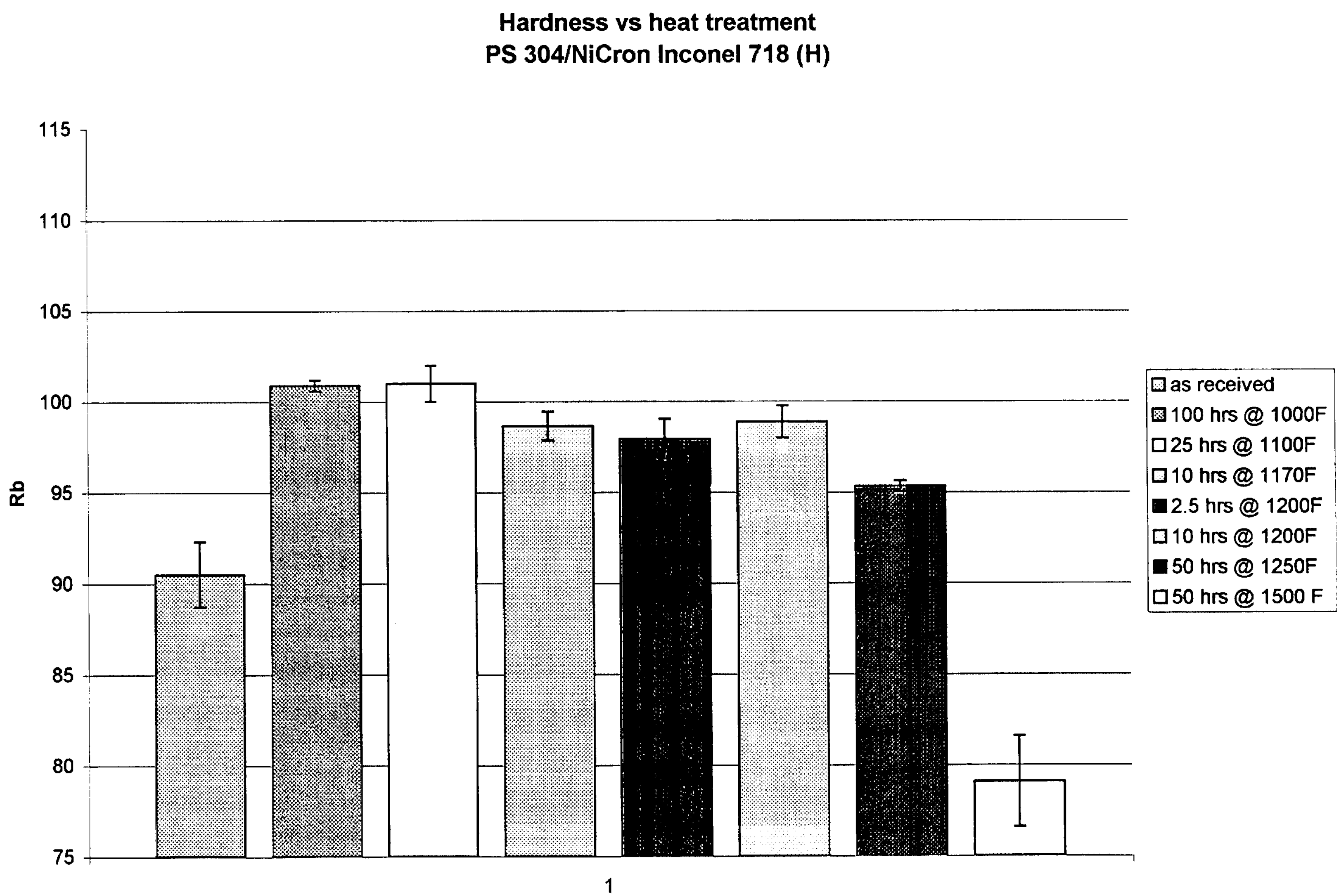

Hardness vs heat treatment
PS 304/NiAl on Stainless 304 (D)
Rb
115
110
105
100
95
90
85
80
75
1
as received
100 hrs @ 1000F
25 hrs @ 1100F
10 hrs @ 1170F
2.5 hrs @ 1200F
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500F


Hardness vs heat treatment
PS 304/NiAl on Inconel x750 (E)
Rb
115
110
105
100
95
90
85
80
75
1
as received
100 hrs @ 1000F
25 hrs @ 1100F
10 hrs @ 1170F
2.5 hrs @ 1200F
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500F


Hardness vs heat treatment
PS 304/NiAl on Inconel 718 (F)
115.0
110.0
105.0
100.0
95.0
90.0
85.0
80.0
75.0
Rb
1
as received
100 hrs @ 1000F
25 hrs @ 1100F
10 hrs @ 1170F
2.5 hrs @ 1200
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500F

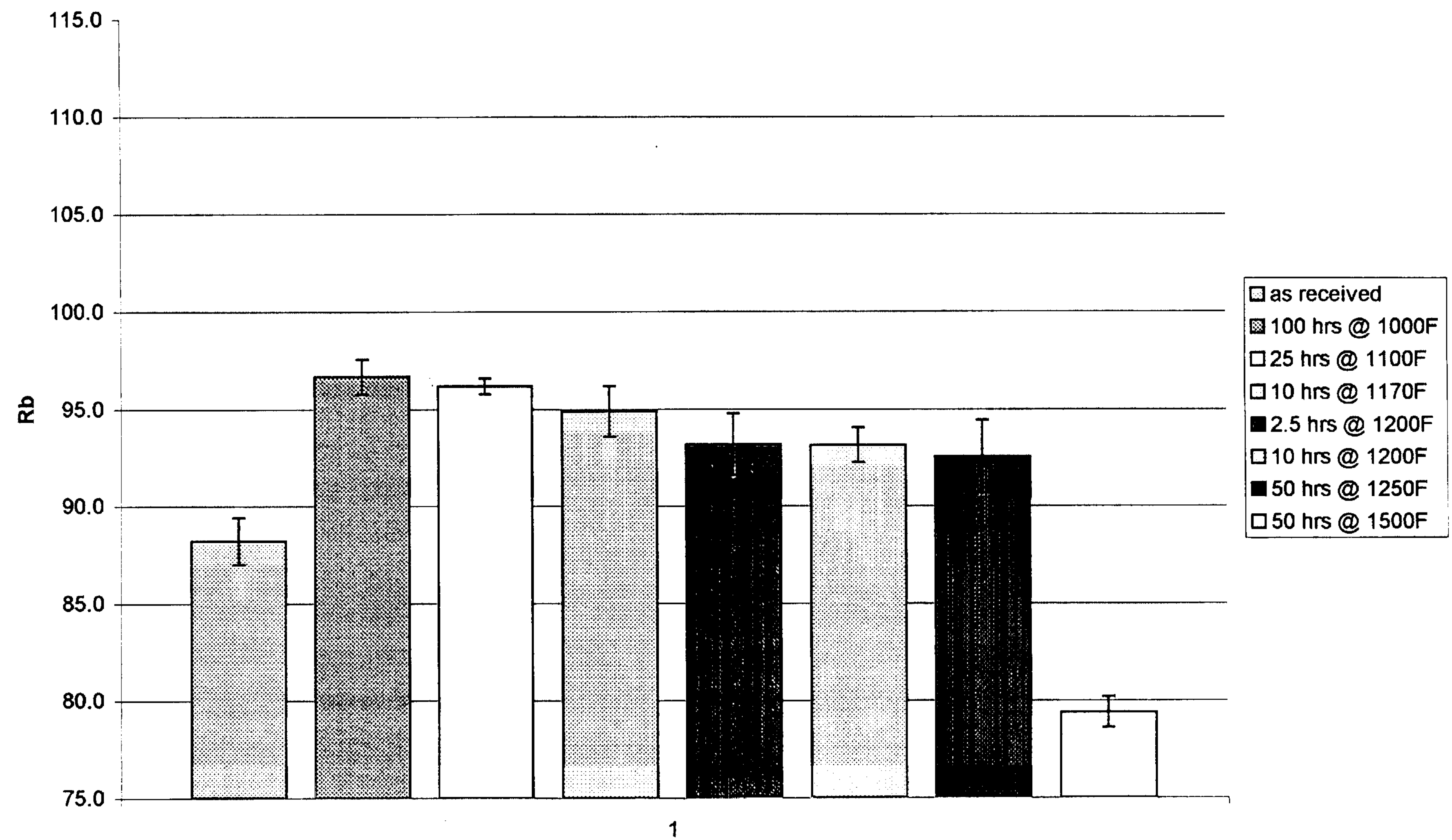
Hardness vs heat treatment
PS 304/NiCr on Inconel x750 (G)

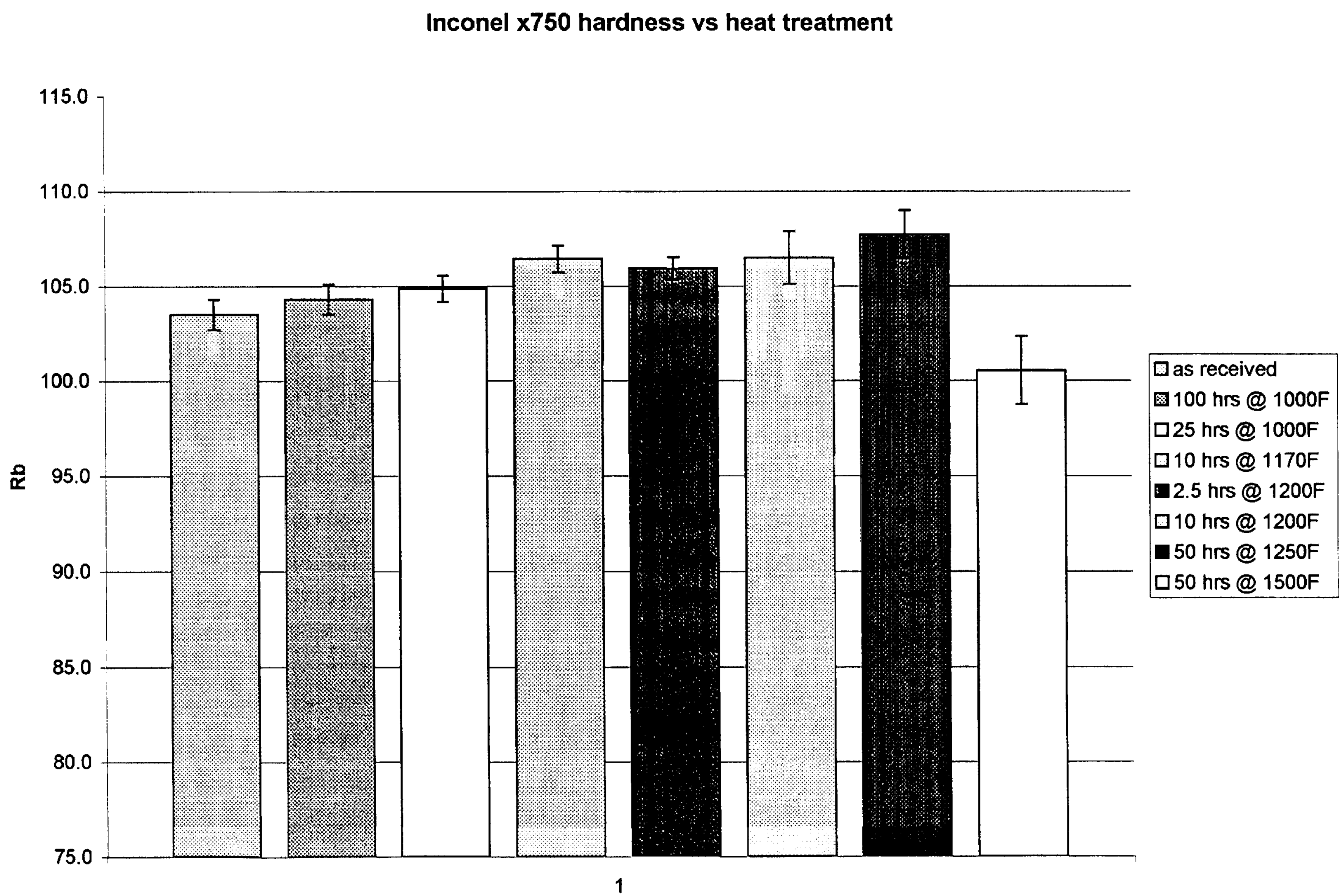




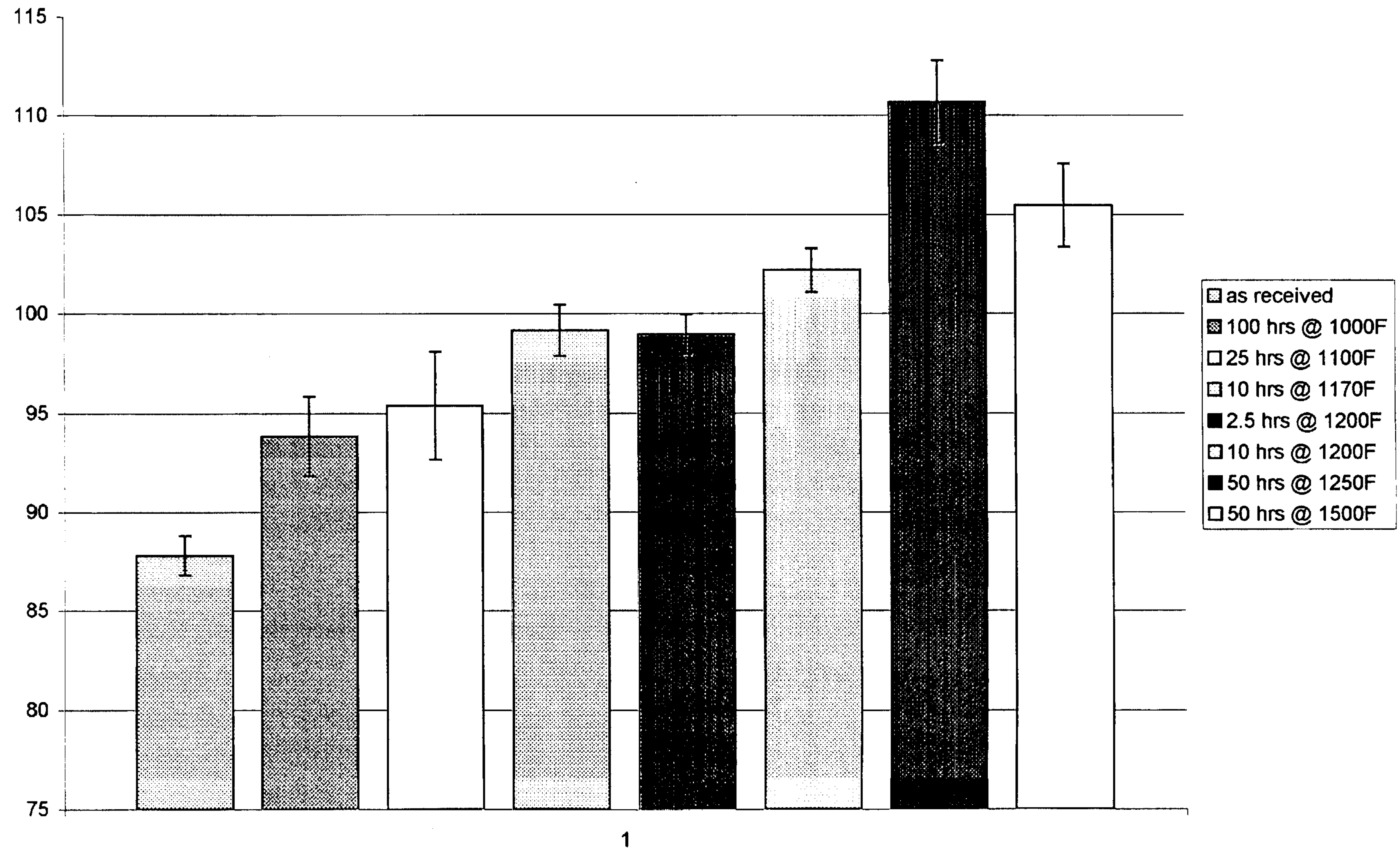
Hardness vs heat treatment
PS 304/NiCron Inconel 718 (H)
Rb
115
110
105
100
95
90
85
80
75
1
as received
100 hrs @ 1000F
25 hrs @ 1100F
10 hrs @ 1170F
2.5 hrs @ 1200F
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500 F

**Hardness vs heat treatment**
**PS 304/NiCr on Stainless 304 (J)**

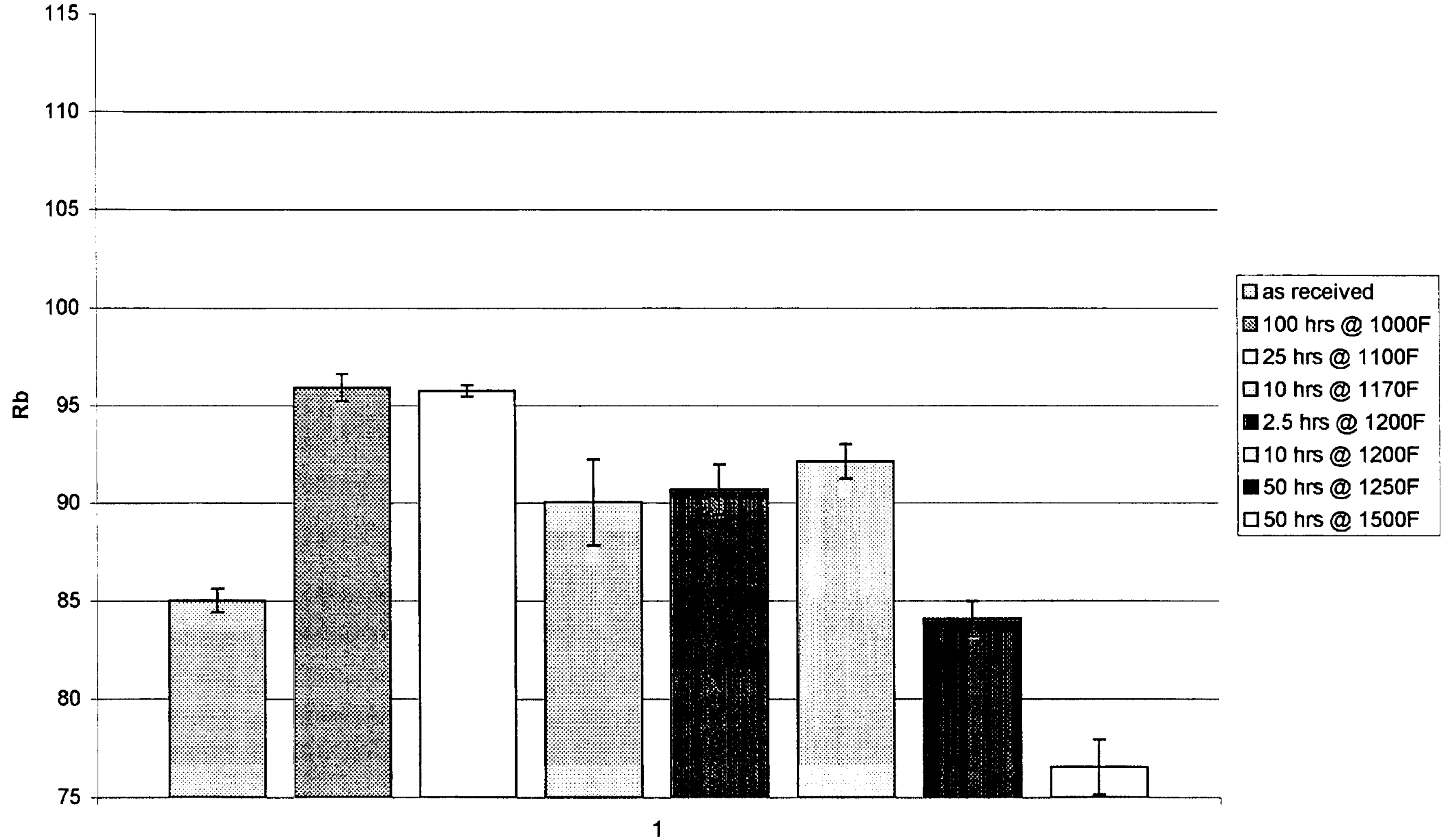



Inconel x750 hardness vs heat treatment
Rb
115.0
110.0
105.0
100.0
95.0
90.0
85.0
80.0
75.0
1
as received
100 hrs @ 1000F
25 hrs @ 1000F
10 hrs @ 1170F
2.5 hrs @ 1200F
10 hrs @ 1200F
50 hrs @ 1250F
50 hrs @ 1500F

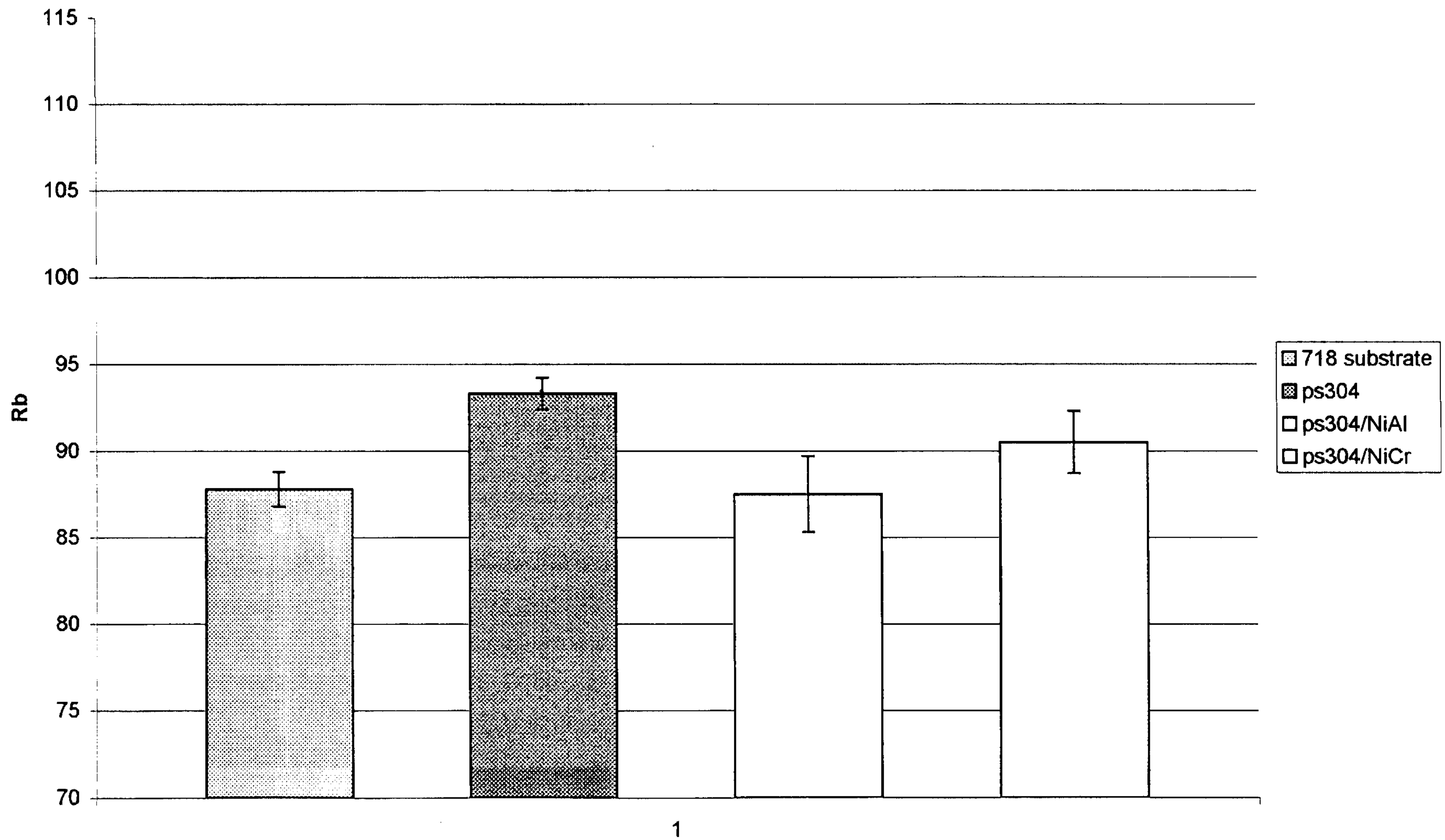
## Hardness of Inconel 718 substrate vs heat treatment



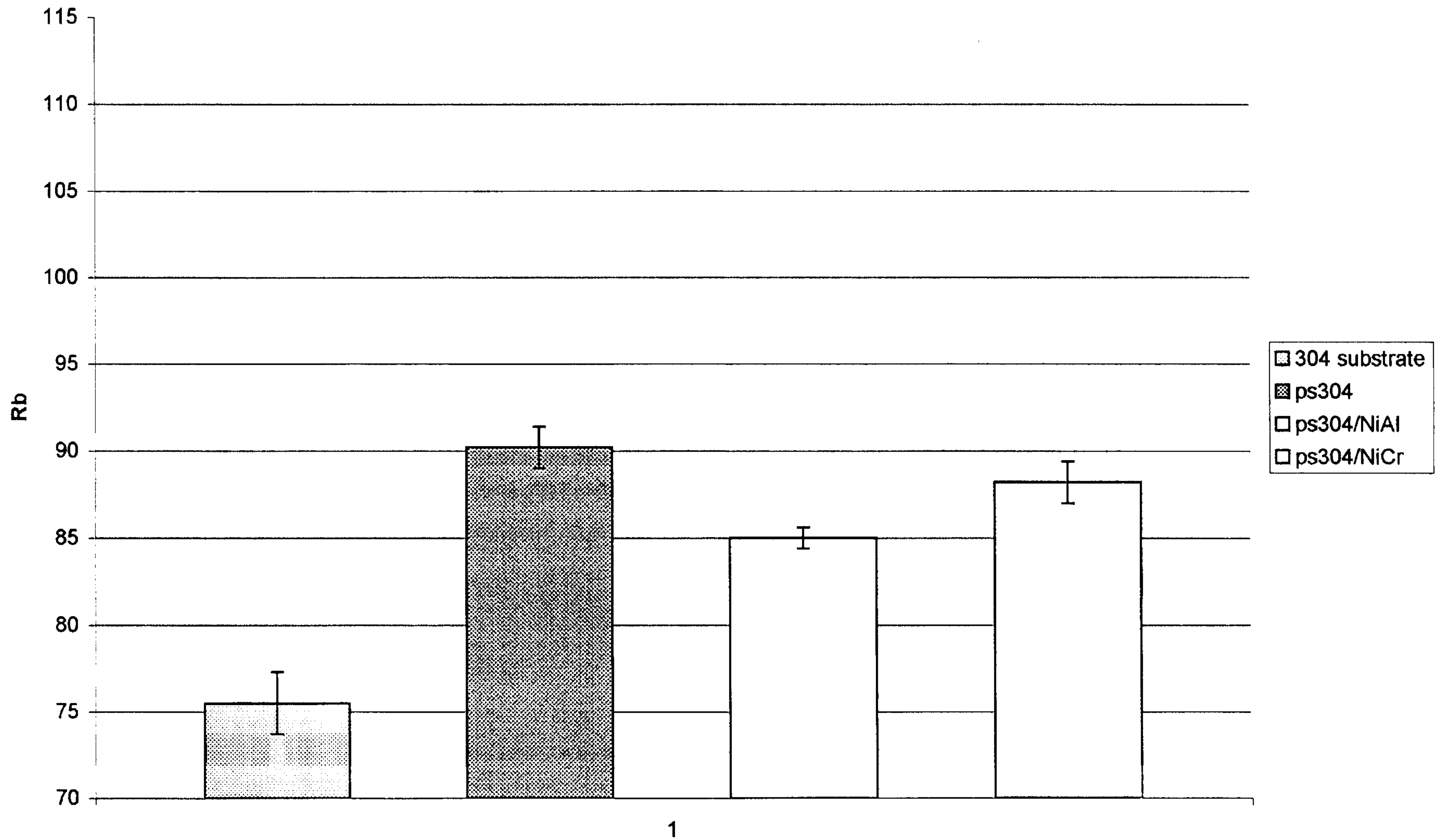
## Hardness vs heat treatment
## PS 304/NiCr on Stainless 304 (D)

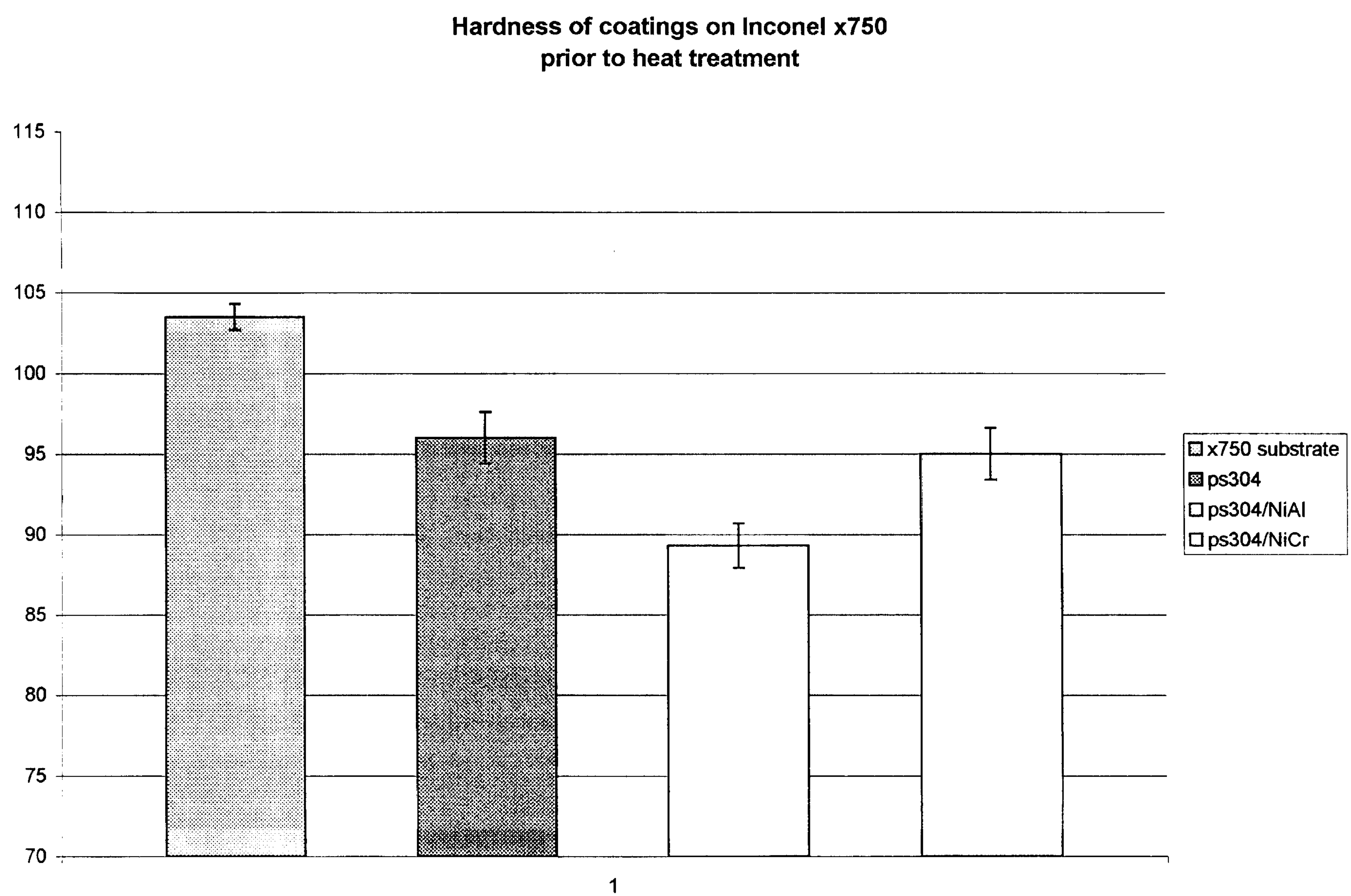


## Hardness of PS 304 coating on Inconel 718 substrate prior to heat treatment



## Hardness of PS 304 coating on stainless 304 substrate prior to heat treatment




Hardness of coatings on Inconel x750
prior to heat treatment
115
110
105
100
95
90
85
80
75
70
1
x750 substrate
ps304
ps304/NiAl
ps304/NiCr

Hardness of coatings on 718 substrate
prior to heat treatment