Heat Treat of 3Z Valve Piston

Federal Manufacturing & Technologies

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Abstract

To improve the processing of 3Z valve pistons in LF7 assemblies, material qualities are being investigated. After a review of the fracture toughness curve, it was noted that a change of a few degrees variation from the 900? F called for by the drawing would affect the fracture toughness of the product. After this observation it was decided to determine how close to the 900? F the parts were heat-treated. The subsequent study indicated that Federal Manufacturing & Technologies (FM&T) equipment held the product at 900? F ? 1.8? F.

Summary

The investigation into the temperatures that the pistons were exposed to during heat treating involved a review of the past heat treat records for both the calibration of the oven and the time and temperature records for the oven control. This led to changes in the drawing callout to specifically calibrate the ovens used at 900? F in addition to the 600? , 750? , 800? , and 1200? F existing calibration points. A designed experiment was performed to determine the effects of part orientation (up or down) in the oven versus the actual temperature the product experiences during the heat treat cycle. The orientation of the part being up or down and location in the oven have no effect on the hardness of the part.

Based on the investigation into the calibration history and the evaluation of the performance of the oven, the calibration organizations believe the ovens satisfied the 900? F requirement and also that the temperature requirements of the 3Z piston heat treat have been met. This is based on the 600? F to 1200? F range of the previous calibrations and the results of placing thermocouples into the actual product.

The thermocouples used have been exchanged for a more accurate type to further improve the process. A subsequent study of the actual part temperature was conducted to ensure the oven accuracy and the actual conditions that the part is exposed to in the oven. Three parts were thermocoupled and placed throughout the oven to study the actual effects of the temperature on the part. The parts held at the 900? F ? 1.8? F. This is well within the ? 10? F allowed.

The results of the calibration and the thermocouple of the parts indicate that all 3Z valve pistons have in the past and will in the future meet the heat treat drawing requirements.

Discussion

This report is a summary of the calibration of the ABAR vacuum heat treat furnaces and the Design of Experiment to observe the effects of location and orientation of the pistons in the oven to ensure the product was processed in accordance with the drawing requirements. The report is divided into two sections--one concerned with the calibration and the other with the effects of heat treating on the part.

Scope and Purpose

This report describes the review conducted to determine if the pistons have been heat treated in accordance with the drawing requirement of 900° F for 4 hours ± 30 minutes.

Activity

Background - Product Definition

The drawing for the 3Z valve piston defines the material as steel corrosion resistant per specification which requires for material up to 4.00 inches diameter, after precipitation hardening at 950° F ± 10° F (510 ± 5° C) for 4 hours ± 0.5 hr, to have the properties shown in Table 1. This material specification replaced the Rocky Flats specification. The drawing requires that the material be heat treated to 900° F ± 10° F for 4.0 hours ± 0.5 hr with the following minimum properties:

The drawing and the material specification differ in the heat treat temperature, tensile strength, yield strength, elongation, and hardness values. Table 2 shows the material properties as received per material heat lot, and Table 3 shows the material properties as tested per each piston lot.

Table 1. Comparison of Rocky Flats Material Specification to the 3Z Valve Piston Drawing

Requirement	Material Specification	3Z Valve Piston Drawing	Difference
Tensile Strength	225,000 psi min	240,000 psi min	15,000 psi
Yield Strength	210,000 psi min	230,000 psi min	20,000 psi
Elongation	10% min	8 % min	2%
Reduction of Area	40% min	40% min	No change
Hardness Rc	47 Rc or equiv	- 44 Rc or equiv	3 Rc

Table 2. Material Properties of As-Received Material

	CarTech 455 Custom Material Specifications		Material Control # Information As Received From Rocky Flats		
			114669	113914	114744
Carbon	0.05	max	0.009	0.01	0.008
Manganese	0.5	max	0.32	0.16	0.2
Phosphorus	0.04	max	0.008	0.008	0.008
Sulfur	0.03	max	0.003	0.002	0.004
Silicon	0.5	max	0.05	0.05	0.024
Chromium	11.00/12.50		11.6	11.6	11.6
Nickel	7.50/9.50		8.1	8.1	8.1
Titanium	0.80/1.40		1.2	1.2	0.97
Columbium and Tantalum	0.10/0.50		0.252	0.262	0.222
Copper	1.50/2.50		2.2	2.3	1.7
Molybdenum	0.5	max	0.02	0.04	0.015
Yield Strength	230	ksi	236.1	239	218
			236.6	239.3	224.8
Ultimate Strength	240	ksi	238.2	229.5	226.2
			240.2	231.5	228.2
Elongation %	8		12.7	10.8	13.4
			13.5	11	17.4
Reduction on Area %	40		53.7	51.8	58.4
			56.2	52.2	59.9
Rockwell "C"	44		47.4	47 to 51	48

Table 3. Material Properties of Heat-Treated Pistons

3Z PISTON DATA							
Material Control # (MC#)	Lot #	Qty tested	Average hardness	Yield strength (ksi)	Ultimate strength (ksi)	% Reduction of area	% Elongation
114669	509	88		242	248	52.1	10.8
				243	249	52.7	11.1
	510			254	260	45.2	8.8
				254	261	42.7	9.9
	511			253	260	47.7	10.9
				253	260	47.8	11.3
	512			253	260	48.0	11.1
				253	260	48.6	10.9
	513			256	262	44.0	no test
				255	261	42.0	10.1
	514			252	258	46.4	10.6
				251	257	48.8	11.0
	515			253	258	44.6	10.6
				250	257	44.5	10.8
	516			250	256	49.2	11.8
				252	259	48.7	11.6
	517			253	259	50.0	11.4
				252	258	49.5	11.9
	518			249	258	46.7	11.0
				251	258	48.9	11.6
	519			253	259	49.7	11.5
				253	259	47.6	11.1
	520			252	259	47.6	11.5
				252	258	44.7	10.6
	521			253	259	45.3	11.2
				252	259	46.4	11.6
	522			250	256	48.9	11.2
				250	256	49.4	11.2
	523			249	256	46.1	10.9
				250	255	49.8	11.0
	524	57		251	257	47.4	11.2
				251	258	47.4	11.4
	525	56		252	258	45.8	10.9
				252	258	45.1	10.8
113914	526	120		242	249	49.0	11.3
				242	249	47.2	10.9
	527	181		245	254	47.3	11.5
				244	251	49.3	11.7
	528	209		245	252	49.5	11.6
				247	252	44.1	10.8
	529	172		238	245	50.0	11.4

The information shown in Figure 1 was abstracted from the figure in Appendix C from Los Alamos National Laboratory (LANL). Figure 1 shows the impact in heat treat temperature on fracture toughness. A change of $\pm 10^\circ\text{F}$ from the nominal 900°F changes the fracture toughness number from ± 30 to ± 50 .

Material Information

- Carpenter Custom 455

$S_y = 230ksi$ $S_u = 240ksi$
 $e = 10\%$ $RA = 46\%$

- Material is annealed followed by age-hardening at 900 F for 4 hours, then air-cooled.

- The 900 F aging produces the lowest fracture toughness of all Custom heat.

Fracture Toughness vs. Aging Temperature

$CVN = 7 - 9T - 40$
 $[K_{IC}]_{min} = 32 - 36ksi \sqrt{in}$

Engineering Sciences &
Applications Division

Los
Alamos

Figure 1. Fracture Toughness Chart Supplied by LANL

Calibration

Background

Parts have been routinely heat treated at AlliedSignal Federal Manufacturing & Technologies (FM&T) for over 17 years to essentially the same calibration procedure. This procedure meets or exceeds industry standards. The calibration of the heat treat ovens is performed in accordance with an internal calibration procedure derived from the requirements of the specification.

This specification is defined through the hierarchical chain:

3Z Piston Drawing

General Requirements

General Requirements, KC Supplement

Temperature Control, Heat Treatment Equipment

Internal Calibration Procedure

The responsible process engineer defines the parameters for the calibration and the types of materials to be heat treated. From these defined requirements, the calibration procedure is developed and then followed by the technician to perform the calibration. The thermocouples used to calibrate the oven are traceable to NIST standards. In the past FM&T has used Ni/Ni/Mo type thermocouples which were calibrated to $\pm 10^{\circ}\text{F}$ with a worst-case accuracy of $\pm 14^{\circ}\text{F}$ based on the RSS method. Since all of the original team members that developed the procedure have retired, it is speculated that this thermocouple was chosen based upon temperature range, availability, durability, and cost. NIST recently changed the accuracy defined for this type of thermocouple, and as a result FM&T will switch to a J type thermocouple for the 650°F and 900°F points. The three ABAR ovens used for heat treatment of product have remained stable as is evidenced by their respective calibration results shown in Tables 4, 5 and 6. Below are the tables containing the calibration data for the past several calibrations of the ABAR furnaces.

Table 4. Past Calibration Results - Oven #1

		ABAR #1					
Temperature in deg. F	4/30/98	2/5/98	1/23/98	7/31/97	2/10/97	8/23/96	3/7/96

Low setpoint	600	600	750	750	750	750	750
Overshoot	7	5	0	0	0	0	0
Operating Temp.	601	601	750	750	750	750	750
Standard Avg.	604	600	748	750	748	750	751
Survey Max.	606	602	750	753	752	755	755
Survey Min.	601	600	748	748	745	745	747
Max. Deviation	5	1	0	3	2	5	5
Min. Deviation	0	-1	-2	-2	-5	-5	-3
Intermediate setpoint	1200	1200	1200	1200	1200	1200	1200
Overshoot	5	0	0	0	0	0	0
Operating Temp.	1202	1200	1200	1200	1200	1200	1200
Standard Avg.	1203	1200	1200	1200	1199	1202	1200
Survey Max.	1208	1205	1205	1205	1205	1207	1205
Survey Min.	1203	1200	1200	1195	1193	1197	1195
Max. Deviation	6	5	5	5	5	7	5
Min. Deviation	1	0	0	-5	-7	-3	-5

Table 5. Past Calibration Results - Oven #2

ABAR #2

Temperature in deg. F	5/16/97	12/6/96
Low setpoint	800	800
Overshoot	0	5
Operating Temp.	800	800
Standard Avg.	801	802
Survey Max.	805	805
Survey Min.	798	800
Max. Deviation	5	5

Min. Deviation	-2	0
Intermediate setpoint	1200	1200
Overshoot	2	5
Operating Temp.	1200	1200
Standard Avg.	1202	1201
Survey Max.	1207	1205
Survey Min.	1197	1198
Max. Deviation	7	5
Min. Deviation	-3	-2

Table 6. Past Calibration Results - Oven #3

	ABAR #3				
Temperature in deg. F	2/20/98	9/2/97	3/18/97	10/2/96	4/16/96
Low setpoint	800	800	800	800	800
Overshoot	0	0	0	5	5
Operating Temp.	800	800	800	800	800
Standard Avg.	800	797	798	799	797
Survey Max.	805	800	800	803	800
Survey Min.	801	795	797	795	795
Max. Deviation	5	0	0	3	0
Min. Deviation	1	-5	-3	-5	-5
Intermediate setpoint	1200	1200	1200	1200	1200
Overshoot	2	5	5	5	5
Operating Temp.	1200	1200	1200	1200	1200
Standard Avg.	1202	1201	1196	1201	1198
Survey Max.	1205	1205	1198	1205	1202

Survey Min.	1201	1197	1195	1197	1195
Max. Deviation	5	5	-2	5	2
Min. Deviation	1	-3	-5	-3	-5

Calibration Summary

Based on the investigation into the calibration history and the evaluation of the performance of the oven, the calibration organizations believe the ovens were capable of meeting the 900° F requirement and also that the 3Z piston heat treat requirements have been met (reference Appendix A, memorandum dated November 9, 1998, and Appendix B, e:mail dated January 5, 1999). In order to ensure calibration at the 900° F temperature in the future, a note has been added to the 3Z valve piston drawing, and this requirement has also been added to the calibration procedure for the oven. The thermocouples used have been exchanged for a more accurate type to further improve the process.

Heat Treat Processing

Background

In the actual use of the part, it would be desirable to have the leading edge of the part hard enough to perform the cutting function as required and the tail or skirt section soft or ductile enough to compress into the piston bore and provide the necessary seal. In order to determine if the orientation of the part (skirt up or down) during heat treat has an effect on the hardness or its ability to withstand cracking, special instructions were provided with production lot 532. A hole was drilled in 4 pistons and a thermocouple was inserted in the hole to determine the time and temperature the product was actually exposed to.

The normal process for heat treatment of the 3Z piston has the following steps in order to comply with the drawing requirement of 900° F for 4 hours ± 30 minutes:

1. Load part into furnace and evacuate chamber to one (1) micron or less.
2. Heat treat to 900 ± 10 deg. F in inert gas and hold 4 hours + 15 - 0 minutes.
3. Gas/fan cool to 121 deg. C (250 deg. F) or below in nitrogen or argon inert gas before unloading from furnace.
4. Detach and submit Time-Temperature chart (to inspection) with the parts.

The ABAR oven (Figure 2) has radial heating elements that run around the inside of the furnace.

Figure 2. Inside of the ABAR Oven

Since the drawing does not define a ramp up or down, the standard practice is to allow the parts to soak at the desired temperature at a rate of 30 minutes per cross-sectional inch of product. This information can be found in AMS 2759/3B Table 4 for Custom 455 material. The 3Z piston is approximately $\frac{1}{2}$ " in diameter. This follows standard practice for the heat treat industry. The oven chart then becomes the record of temperature versus time required. The chart is compared to the drawing requirements to ensure the correct time at temperature was accomplished.

In order to determine the effects of heat treating the parts, an experiment was designed to study the various effects of orientation and location within the oven. The effects studied included the orientation of the parts as related to hardness, the actual temperature the part sees internally to ensure part uniformity, and how accurate the controller is on the oven.

The heat treatment of Lot 532 (103 parts) was performed according to the usual instructions and included the following special instructions:

1. The lowest 51 control numbers were placed with the piston skirt placed down and evenly distributed in all trays and in the oven.
2. The highest 52 control numbers were placed with the piston skirt placed up and evenly distributed in all the trays and in the oven.
3. Four reference units were provided by engineering with a thermocouple wire attached, and one was placed in the center of the oven (calibration point) with the skirt up and one adjacent to it with the skirt down. The other two were placed within the tray and as far from the center as possible, one with the skirt up and the other with the skirt down (Figure 3).
4. The location of each control number was recorded, showing its position in the tray (Figure 4) and the location of each tray in the oven. This included the reference units also.
5. A total of eight (8) additional tensile samples were provided and placed evenly throughout the trays with the parts. Each sample was identified with a serial number on the end and its respective location recorded for both positions in the tray and in the oven.

Figure 3. Parts Placed With Skirt Up and Down

The thermocoupled pistons were evenly spaced throughout the oven to determine the actual part temperature. The hole was drilled into the middle of the piston. The end of the thermocouple was inserted into this hole (Figure 5).

Figure 4 shows the serial number of the pistons as they were inserted into the oven for Lot 532. The special instructions provided to processing precede this chart.

07 03

06

01

04 02

OVEN DOOR

Units with the skirt down Additional tensile specimens

Units with the skirt up Production tensile specimens

Thermocouple sample parts

Figure 4. Orientation of Pistons in Oven

Figure 5. 3Z Valve Piston With the Thermocouple Attached

The parts were heat treated in the ABAR furnace to the 900° F requirement and held there for approximately 4 hours and 15 minutes, according to the oven temperature chart. The parts were then all tested for hardness (Rc). Results are shown in Figure 6.

Figure 6. Results of the Hardness Testing. (Values shown are Rc hardness.)

Each piston was measured for hardness on the flat area near the skirt end.

The data below lists by serial number the hardness of the 103 parts.

1	48.3	102	50.9	20	52.2	31	53.6
46	48.9	50	51.0	89	52.2	39	53.6
112	49.0	59	51.0	116	52.4	88	53.6
17	49.3	86	51.0	73	52.5	58	53.7
93	49.4	122	51.0	12	52.6	18	53.8
111	49.7	52	51.1	28	52.6	36	53.8
125	49.9	57	51.2	119	52.6	10	54.0
127	50.1	98	51.2	83	52.7	42	54.0
91	50.3	113	51.2	3	52.8	126	54.0
101	50.3	110	51.4	47	52.8	71	54.1
123	50.4	100	51.5	65	52.9	43	54.2
99	50.5	77	51.6	72	52.9	34	54.6
108	50.5	13	51.7	87	52.9	49	54.6
120	50.5	29	51.7	15	53.2	79	54.7
63	50.6	9	51.8	26	53.2	90	54.7
103	50.6	54	51.8	51	53.2	69	55.2
55	50.7	61	51.8	56	53.2	118	55.5
94	50.7	84	51.8	70	53.3	21	55.6
109	50.7	104	51.8	4	53.4	33	55.6
97	50.8	68	51.9	8	53.4	115	56.1
106	50.8	121	51.9	23	53.4	67	56.8
124	50.8	6	52.0	22	53.5	74	56.9
53	50.9	40	52.0	27	53.5	82	56.9
81	50.9	7	52.1	44	53.5	41	57.6
92	50.9	62	52.1	24	53.6	32	58.1
96	50.9	16	52.2	25	53.6		

The statistical hardness data from Lot 532 is as follows:

The data is hardness in Rockwell C (Rc) units.

MAX	58.1
MIN	48.3
AVG	52.4
STDDEV	1.9

Two of the tensile samples were sent to the materials lab for evaluation of the physical properties. The parts were tested in accordance with a Laboratory Test Request (LTR). Table 7 contains the results of the tests performed:

Table 7. Laboratory Test Results for Design of Experiment

Sample	Mechanical Properties			
	Yield	Ultimate	Elongation	Reduction of Area
	KSI	KSI	%	%
1	240	240	12.4	52.5
2	235	241	12.1	52.6
Average	234	240.5	12.3	52.6
3Z Valve Piston Req't	230 min	240 min	8 min	40 min

It should be noted that this material lot (532) just barely meets the requirements for yield and ultimate strength. As noted earlier in Table 3, the material control lots prior to 114744 had significantly higher values.

Actual Part Temperature in the Oven

In accordance with the special instructions, four pistons were drilled in the side of the part to allow for a thermocouple to be installed. The thermocouple selected had an uncertainty of $\pm 2^\circ\text{F}$. The four units were wired and placed in the oven as shown in Figure 7.

Figure 7. Parts Thermocoupled to Provide Actual Part Data

The results are shown in Table 8. One of the thermocouples did not provide any results to the event recorder during the test. The other three are shown, and their respective location can be determined from Figure 4. The results indicate the part temperatures were within the drawing-specified requirements. The thermocouple was located in the center of the product as shown in Figure 5. The stability of the oven is clearly demonstrated by the very small changes in temperature during the cycle. The oven chart indicates the parts were @ 900°F for 4 hours 15 minutes which is different than the 3 hours 45 minutes indicated at the center of the part. Both are within the drawing-specified time and temperature. The shaded areas indicate the start and stop of the $900 \pm 10^\circ\text{F}$ zone. A typical oven chart is shown in Figure 8.

Table 8. Thermocouple Results From Actual Product

TIME	Piston # 114	Piston # 78	Piston # 14
7:09:44 AM	85.9	85.8	85.9

7:14:44 AM	85.9	85.8	85.8
7:19:44 AM	488.2	688.4	483.5
7:24:44 AM	685.6	751	713.3
7:29:44 AM	776.3	794.7	805.6
7:34:44 AM	833	845.2	851.8
7:39:44 AM	862.5	870.3	874.4
7:44:44 AM	877.2	881.9	884.8
7:49:44 AM	885.2	888.3	890.3
7:54:44 AM	890.1	892.6	893.7
7:59:44 AM	893.5	895.4	896.1
8:04:44 AM	895.7	897.5	897.7
8:09:44 AM	897	898.6	898.6
8:14:44 AM	898	899.4	899.3
8:19:44 AM	898.5	899.9	899.6
8:24:44 AM	899.1	900.4	900.1
8:29:44 AM	899.3	900.6	900.3
8:34:44 AM	899.4	900.7	900.4
8:39:44 AM	899.5	900.7	900.4
8:44:44 AM	899.5	900.8	900.5
8:49:44 AM	899.5	900.8	900.5
8:54:44 AM	899.5	900.8	900.4
8:59:44 AM	899.5	900.8	900.4
9:04:44 AM	899.5	900.8	900.4
9:09:44 AM	899.5	900.8	900.4
9:14:44 AM	899.5	900.9	900.3
9:19:44 AM	899.5	900.8	900.3
9:24:44 AM	899.5	900.8	900.4
9:29:44 AM	899.5	900.8	900.4

9:34:44 AM	899.4	900.8	900.3
9:39:44 AM	899.4	900.8	900.3
9:44:44 AM	899.5	900.8	900.3
9:49:44 AM	899.4	900.8	900.3
9:54:44 AM	899.4	900.8	900.3
9:59:44 AM	899.4	900.8	900.3
10:04:44 AM	899.4	900.8	900.3
10:09:44 AM	899.4	900.8	900.3
10:14:44 AM	899.4	900.8	900.3
10:19:44 AM	899.4	900.7	900.3
10:24:44 AM	899.3	900.7	900.2
10:29:44 AM	899.3	900.8	900.2
10:34:44 AM	899.2	900.7	900.2
10:39:44 AM	899.3	900.7	900.2
10:44:44 AM	899.3	900.7	900.2
10:49:44 AM	899.4	900.8	900.3
10:54:44 AM	899.3	900.8	900.2
10:59:44 AM	899.4	900.6	900.2
11:04:44 AM	899.3	900.7	900.2
11:09:44 AM	899.3	900.7	900.1
11:14:44 AM	899.2	900.6	900.1
11:19:44 AM	899.4	900.8	900.3
11:24:44 AM	846.6	792.2	825.2
11:29:44 AM	189	234.5	217.9
11:34:44 AM	152.8	162.5	160.6
11:39:44 AM	141.2	144	143.7
11:44:44 AM	136.8	137.6	137.6
11:49:44 AM	135.1	135	135.3

11:54:44 AM	134.3	134	134.3
11:59:44 AM	134.2	133.7	134.1
12:04:44 PM	134.2	133.6	134
12:09:44 PM	134.2	133.6	134
12:14:44 PM	134.3	133.7	134.1
12:19:44 PM	134.3	133.7	134.1
12:24:44 PM	134.5	133.9	134.3
12:29:44 PM	134.6	134	134.4
12:34:44 PM	134.6	134	134.4
12:39:44 PM	134.7	134.1	134.5

Typical Oven Chart

Figure 8. Typical Oven Chart

Accomplishments

The results of the thermocouples on the parts indicate the parts were heated to 900° F with a standard deviation maximum of 1.8° F for the three thermocouples. This, compared with the results of past calibrations, provides results that the 3Z pistons have been and are currently being heat treated to the drawing requirements. The material control lot 114744 currently in use only meets the minimum requirements after heat treat. The chemistry of the lot does show a difference from past lots and can contribute to changes in mechanical properties being different. Lot 533 was the next lot heat treated to the same temperature conditions, and the ultimate tensile strength was 238 ksi or 2 ksi ULL of the required 240 ksi minimum.

The calibration changes necessary to ensure that the oven is capable at 900° F have been put into place. This will ensure a method of compliance to the drawing by calibrating specifically at the 900° F temperature. The following suggestions could be implemented to improve the manufacturability of the product:

✍ A drawing change to:

- Lower 240 ksi minimum requirement.
- Change to UTS average requirement instead of minimum.
- Change heat treat to 950° F – 4 hrs for more uniformity and toughness.

✍ Change Steel Corrosion Resistant per specification to bring it in line with drawing requirements.

- Change heat treat callout to 900° F – 4 hrs instead of 950° F – 4 hrs.
- Change UTS and YS callouts to 240 ksi and 230 ksi minimum.
- Change Ti content to 1.2/1.4 %.
- Change Cu content to 2.0/2.5 %.

NOTE: The material supplier implied that such changes would not increase the cost significantly.

✍ Study the effects of changing the heat treating process to a 950° F – 4 hr cycle on product performance. Potential improvements are:

- More uniform mechanical properties,
- Better fracture toughness and less cracking,
- Reduced dependence of mechanical properties on chemistry variations.

If future lots of pistons are to be produced from material control lot 114744, the above items need to be considered and drawings changed accordingly to ensure that the heat treated material can meet the drawing requirements.

Appendix A

Memorandum, 3Z Piston Heat Treat Operations

Memorandum
Federal Manufacturing & Technologies

Date: November 9, 1998
To:
From:
Subject: 3Z Piston Heat Treat Operations

The purpose of this memo is to provide the results and a summary of the investigation into the calibration of the Abar vacuum furnace that is used to perform the heat treat operation on the 3Z pistons. The investigation into the calibration of the vacuum furnace was initiated after a cracking phenomenon was noticed on some 3Z Pistons during assembly operations. In your earlier e-mail you asked me to answer two questions.

1. Did we meet the 3Z piston drawing requirement, Note 3 (vac heat treat @ 900 ± 10 degrees F) on all previous lots?

Answer: Yes, based upon our investigation into the calibration history and an evaluation of the performance of the oven, the calibration organizations believe the oven was capable of meeting 900 ± 10 degrees F, and believe to the best of our abilities that the temperature requirements of the 3Z piston heat treat were met

2. Can we meet this drawing requirement on future lots.

Answer: Yes, we believe the vacuum furnace calibration process in conjunction with its proper operation will ensure that we continue to meet the requirements of the 3Z piston drawing.

Summary

In short the calibrating organizations believe the Abar vacuum furnace used to perform the heat treat operation on the 3Z pistons was capable of meeting the 900 degree F requirement. However, due to the criticality of the heat treat temperature we believe that modifications to the existing calibration process are warranted. To this end, a calibration point at 900 degrees F has been added to the calibration process and thermocouples with a tighter calibration certification have been selected for the 900 degree

Background

Although I cannot speak for the heat treat process, a review of past calibration data and a performance evaluation of the vacuum furnace suggests that the furnace was more than capable of meeting the 900 ± 10 degree requirement. The calibration organizations were not aware of the criticality of this heat treat process to the function of the 3Z piston.

The calibration process for the vacuum furnace was developed approximately 17 years ago and was based upon the requirements. Until recently, Ni/Ni/Mo thermocouples calibrated to ± 10 degrees F were used to certify the heat treat furnace over the range of 750-2100 degrees F. The furnace was calibrated at three points over this range (750, 1200, 2100). Normal calibration practice is to use calibration standards that are certified four times tighter than the instrument being calibrated. In this instance the four to one ratio between the standard and the furnace readout was not maintained. Since all of the original members of the team that developed the calibration procedure have retired, I can only speculate that these thermocouples were chosen based upon temperature range, availability, durability and cost. Thermocouples meeting the four to one ratio over the entire calibrated range of the furnace cost around \$100.00 per inch. In addition, heat treated material is accepted based upon its ability to meet certain material property tests after the heat treat operation. Heat treated material has continued to successfully pass all material property testing until the recent problems with the 3Z piston.

The recent problems with the 3Z piston have resulted in a thorough evaluation of vacuum furnace performance and the calibration procedure. Even though the four to one ratio was not maintained between the standard and the furnace readout, an evaluation of the furnace at 900 degrees F using type "J" thermocouples (± 2 degrees F) suggests that the oven is extremely stable at that temperature. It also suggests that the temperature distribution across the working surface of the furnace is very good (± 2 °F).

Due to the criticality of the heat treat to the performance of the 3Z piston, the calibration organizations are investigating incorporating some enhancements to the calibration process. First, a calibration point has been added at 900 degrees F. This will assure the furnace readout accuracy at the temperature of interest to the 3Z piston. In addition, we are using type "J" thermocouples certified to ± 2 °F to perform the furnace calibration points at 650 and 900 degrees F and the Ni/Ni/Mo thermocouples above 900 degrees F. This process is extremely time consuming and adds two to three days to the calibration of the furnace. The calibration organizations are currently investigating the use of a platinum sheathed platinum rhodium thermocouple that is capable of meeting the four to one ratio over the entire operating range of the vacuum furnace. These thermocouples are extremely expensive (approximately \$100.00/inch), but would reduce the overall calibration time by two to three

Appendix B

Memorandum, Re: Results of the Investigation into the ABAR Vacuum Furnace Calibration

Subject: Re: Results of the investigation into the ABAR Vacuum furnace calibration

Here are the answers to your questions.

- 1. What happened to the oily/dirty thermocouples that were replaced? Answer: The thermocouples and the readout were taken out of service, but are still with the strip chart recorder.
- 2. If we were not at 4 times where approximately were we? Answer: We were at a 1 to 1 ratio. According to [REDACTED], this would equate to a worst case condition of ± 14 degrees for the vacuum furnace using the RSS method. This calibration method has been in effect for approximately 17 years. The people that established the process are no longer here. Generally, mechanical properties are tested for heat treated material to verify proper heat treat.
- 3. What is the uncertainty number that relates to the 900 degree F reading indicated by the oven chart. Answer: In the past we believe that it is was most likely ± 14 degrees [REDACTED]. The recent calibration was performed using thermocouples certified to ± 2 degrees F. This would provide a 5 to 1 ratio to the ± 10 degree requirement at 900 degree F.
- 4. According to the calibration results [REDACTED] it shows a total deviation of approximately ± 5 degrees, does this 10 degrees add to the uncertainty. Check the 7/31/97 and 2/10/97 dates for examples CE 51030. Answer: It is used as a check to make sure the oven is performing within the ± 10 degree limit to which it is certified. This deviation is factored into the ± 14 degree F referenced above.
- 5. The calibration results indicate the furnace was calibrated at 600, 750, and 800 degrees F and not just 750. Why were we changing around? Answer: It is my understanding that there are at least three ABAR vacuum furnaces that can be used to heat treat 3Z pistons [REDACTED]. The calibration points were selected by the heat treat process engineer depending upon what is processed in the furnace. [REDACTED] and [REDACTED] have a lower limit of 800 degrees F. [REDACTED] had a lower limit of 750 degrees F until the process engineer requested that the lower limit be changed to 600 degrees F so they could heat treat some beryllium copper material. Calibrations points and limits can and are sometimes changed by the process engineer when special requirements exist.

I hope this has answered your questions. If you should have further questions just let me know.

Appendix C

LANL-Supplied Chart for Fracture Toughness

Material Information

• Carpenter Custom 455

$$S_y = 230ksi \quad S_u = 240ksi$$

$$e = 10\% \quad RA = 46\%$$

• Material is annealed followed by age-hardening at 900°F for 4-hrs, then air-cooled.

• The 900°F aging produces the lowest fracture toughness of all Custom heats.

$$CVN = 7 - 9ft - lbs$$

$$[K_{IC}]_{CVN} = 32 - 36ksi\sqrt{in}$$

Engineering Sciences & Applications Division

Los Alamos