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Furnace Atmosphere Analysis by the Shim Stock Method

In the August 2004 issue of IH, we learned that carbon potential of a furnace atmosphere is reliably and accurately measured using a shim stock or carbon gradient bar test. The test involves a relatively simple procedure, but it can be complicated and provide misleading information if not performed properly. The following guidelines will help ensure that the results obtained are reliable.

Shim stock requirements

Shim stock should be from 0.004, 0.010 or 0.015 in. (0.10, 0.25 or 0.38 mm) thick very low carbon annealed sheet (e.g., AISI 1005, 1008 or 1010). Test coupon size depends on the analysis method employed (Table 1). Common sources of error are due to oils, grease, or dirt on the coupons, so care should be taken in handling them, such a the use of rubber gloves. Shims should not be cleaned with solvent if the combustion method is to be used, but instead clean the surface using grit blasting or other abrasive techniques if necessary. Coupons can be cleaned with solvent (e.g., alcohol or acetone), rinsed with water, wiped clean and dried if using the weight-gain method.

Shim stock test methods

In the combustion method, three shims are typically hung from a rod and inserted into the furnace through the furnace wall or roof. The insertion depth in the following table must be adequate to allow furnace atmosphere to circulate freely around them and to position the shim in a location representative of the load in the furnace, typically 2 to 3 in. (50 to 75 mm) from side of the workload (batch equipment) or 2 to 3 in. above the work (continuous equipment).

	Min. depth, in. (mm)
Batch furnace	4-7 (127-178)
Continuous furnace	7-10 (178-255)

The shims are pulled from the furnace after adequate dwell time (see following table) and quickly water quenched or slow cooled in a similar manner to the weight-gain method described below.

Temperature, °F (°C)	Austenitizing time, min
1550 (845)	45-65
1600 (870)	30-50
1650 (900)	20-40
1700 (925)	15-30
1750 (955)	10-25

In the weight-gain method, shims are weighed and loosely wrapped and secured around a rod before inserting into the furnace thorough a specially constructed shim puller arrangement (Fig. 1). The insertion depth and location requirements are the same as for the combustion method.

After adequate dwell time, the shims are pulled from the furnace and cooled for an adequate time (Table 2) in a black body environment surrounded by furnace gas or nitrogen. Typically, the shim is pulled to a position between the valve and end cap (Fig. 1).

Shim stock analysis

The combustion method for carbon (or carbon and nitrogen) determination is the preferred test method and is the referee if different analysis methods yield different results. A sample weight of ~1 g is placed in a carbon determinator, which should be calibrated against standards for expected %C, if known. All three shims run should be analyzed, using the average of all readings. If one of the three readings varies significantly (> 4-5 points), it should be discarded, using an average of the other two. If two of three readings are significantly different, a new set of samples should be run.

In the weight-gain method, the difference in shim weight before and after exposure to the furnace atmosphere is determined using a highly accurate analytical balance. The carbon potential of the atmosphere is calculated using the following equation:

$$C_p = \frac{(Wg \times 100)}{W_f} + \%C_o$$

where C_p is the carbon potential of the atmosphere, W_g is the weight gain (the difference in before and after), W_f is the final weight of the test coupon and C_o is the original carbon content (wt%) of the test coupon.

Shim stock analysis determines the maximum carbon potential of the furnace atmosphere up to the limit of carbon saturation in austenite shown in the following table.

Max. carbon solubility in austenite Temp. , °F (°C)	Theoretical saturation limit, %C
1550 (845)	1.05
1600 (870)	1.11
1650 (900)	1.20
1700 (925)	1.33
1750 (955)	1.40

Table 1 Shim stock size

Analysis method	Test coupon size Surf. area, in² (cm²)	Strip size, in (mm)	Remarks
Weight gain	3.75-5.5 (25-35)	1.25 × 3 (30 × 75) or 1.375 × 4 (35 × 100)	1/8 in. (3 mm) diam. punched holes in each end sometimes used to wire shim to rod.
Combustion	2.25 (15)	1.5 × 1.5 (38 × 38)	1/4 in. (6 mm) diam. punched hole, ~0.250 in. (6 mm) from edge for handling

Generally, shim carbon content determined by these methods is higher than the surface carbon of the parts (due to factors including mass, alloying elements, diffusion characteristics, etc.). Exceptions are steels that contain high percentages of carbide forming elements; these steels have higher surface carbon than the shim stock.

The shim stock test also is applicable to carbonitriding because the weight gain from the adsorption of both carbon and nitrogen from the furnace atmosphere may be treated as though it were carbon alone; that is, the same calculations are used as those for a carburizing atmosphere.

Precautions

The following precautions should be noted:

1. Always expose the shim to a representative sample of the furnace atmosphere (away from areas such as atmosphere inlets and heating sources) and place it in an area of unobstructed flow. To correlate the shim to other atmosphere analysis devices (oxygen probe or infrared analyzer), place it in close proximity (6 in., or 150 mm) to the device or sample port.
2. Expose the shim to the furnace atmosphere for a time sufficient to allow saturation to the limit of carbon in austenite for the temperature selected. Insufficient exposure produces a carbon gradient in the part resulting in a lower average value. Overexposure may produce massive carbides resulting in excessively high readings.
3. Place the shim in an area where the shim temperature and the temperature of the surrounding furnace are within acceptable limits (preferably ±10°F).
4. The furnace being tested should be at temperature and the furnace atmosphere stabilized.
5. Avoid decarburizing the shim and obtaining a false (low) reading if cooling the shim in puller assembly; cool under nitrogen or using the furnace atmosphere (Caution: sooting must be avoided).
6. For continuous furnaces, the carbon potential indicated by the shim represents only the zone or area tested and may be influenced by adjacent zones or areas.

Table 2 Minimum shim cooling time for weight-gain method [3]

Temperature, °F (°C)	Dwell time, min for different shim thickness		
	0.004 in. (0.10 mm)	0.010 in. (0.25 mm)	0.015 in. (0.38 mm)
1550 (845)	89	353	450
1650 (900)	56	156	274
1750 (955)	37	101	175

Shim appearance

A properly exposed and cooled shim should have a particular appearance. Shims containing 1.30% carbon or less should be bright and clean (scale free). A slightly oxidized or discolored surface indicates an improper technique. The weight-gain method produces a slightly high reading because of the increase in weight of the oxide.

Shims containing approximately 1.40% carbon or greater have a dull matte gray finish. Depending on the actual carbon content, the shim surface may be coated with soot, which is a normal appearance caused by an atmosphere of high carbon potential. These deposits should be blown or wiped off prior to weighing the shim.

Final thoughts

The shim stock analysis method is highly accurate provided the proper methods are followed. Shim stock analysis is important in establishing a proper carburizing process and correlating furnace atmosphere control instrumentation with the actual carbon potential of the furnace atmosphere. It is highly recommended that shim stock results be correlated with actual workpieces or the analysis of turn bars so as to fully understand the behavior of the furnace atmosphere and to arrive at an optimized set of operating conditions. **IH**

Bibliography

1. ASM Metals Handbook, Vol 4, ASM International, p 589-590, 1991
2. T.H. Lotze, *Shim Stock Analysis,* Tech. Datasheet T4408, Super Systems Inc., Feb. 2001
3. Ensure Atmosphere Carbon Potential, *Heat Treating,* April 1993

Additional related information may be found by searching these (and other) key words/terms via BNP Media LINX at www.industrialheating.com: carbon potential, shim stock test, carburizing atmosphere, carbon determinator, oxygen probe, infrared analysis, sooting

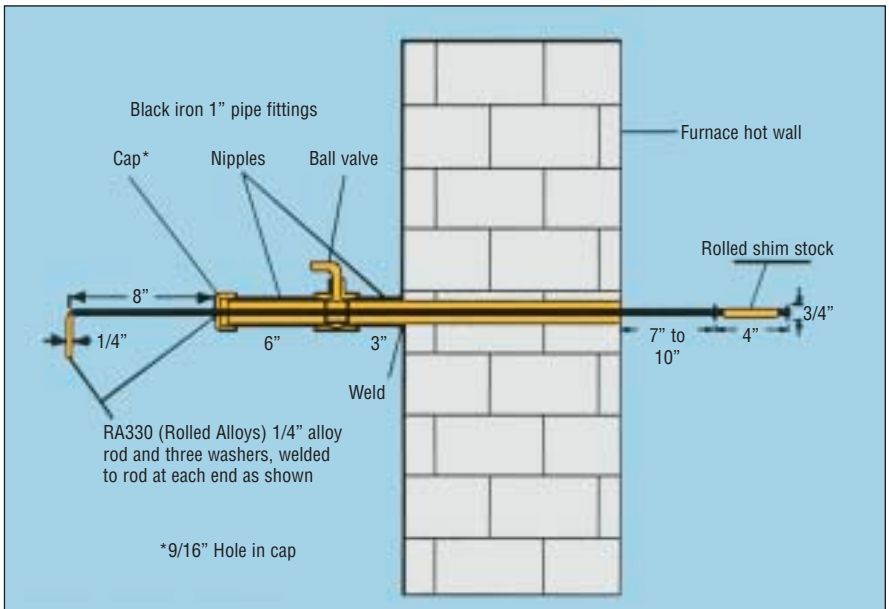

Fig 1 Typical shim-puller arrangement. Courtesy of Super Systems Inc.