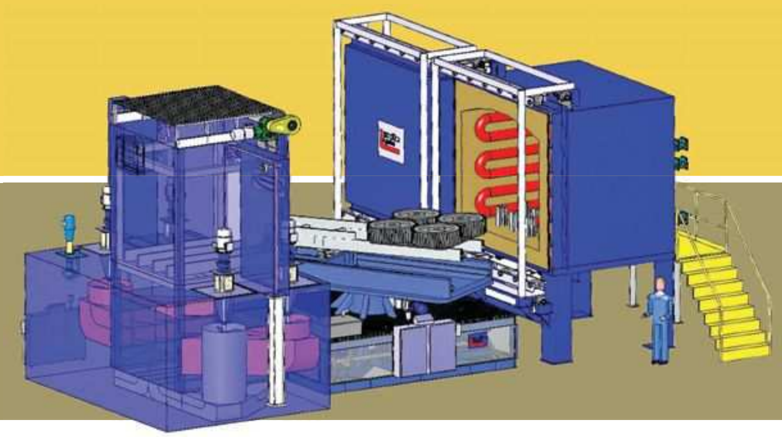
Intergranular Oxidation: Scourge or Does it Really Matter?



Wallace (Jack) Titus – AFC-Holcroft; Toledo, Ohio

Endothermic gas carburizing and liquid quenching will be with us for probably another 50 years at least. Why? Because, for the money, there just is no other case-hardening process that can elevate the properties of such a wide range of ferrous alloys.

Intergranular oxidation (IGO) can be a byproduct of gas/atmosphere carburizing. Is IGO really the scourge of endo carburizing as some believe, or does IGO really matter in the end?

Atmosphere Carburizing

Historically, when the water-gas reaction entered our understanding after endothermic gas became the primary carburizing agent, the water vapor by dew point was the primary measurable because infrared analyzers had yet to be invented. Eventually, as automatic dew-point controllers became the size of bookcases, infrared analyzers began their acceptance into the heat-treat industry. Primarily as complements to the oxygen probe, CO_2 infrared analyzers – with their zero and span calibration gases – displaced dew point and still can be found in operation today. Oxygen probes (in situ or external) have become the primary control strategy, mainly because they provide a near fool-proof and easy calibration method – shim stock analysis – the old standby.

We can control surface carbon to +0.05%, case carbon profiles to +0.005 inches (0.125 mm) and core hardness to within the Rockwell "C" calibration block's standards. Lingering in the background, however, the phenomenon called IGO is seen to be uncontrollable and will doom gears and other products to premature failure. But will it really?

IGO: In the Beginning

Ever since it was discovered, probably with the introduction of optical metallography over 70 years ago, IGO (Fig. 1) quickly became a possible explanation for all manner of product failures. Organizations primarily involved in gear applications are especially focused on establishing limits to IGO depth, such as the standards or guidelines outlined in the AGMA standard 923–B05 item 16.1. The IGO depth is characterized according to gear grades 1, 2 and 3 relating to the ECD (effective case depth) and also with the existence and distribution of iron carbide (Fe_3C).

The gear-tooth root, specifically 60 degrees from the root midpoint, usually sustains the highest bending stress. As a result, it has become the target for IGO specifications, primarily because it is too expensive to precision grind (among other complications). The tooth face, which must survive the sliding and compressive forces, is routinely ground in most drive-train applications. This grinding operation removes any IGO.

Eliminating IGO is not necessarily the primary reason for face grinding, which re-establishes the tooth profile after quenching because gears can only function properly when the loading is distributed uniformly over the tooth contact area. With or without the presence of IGO and regardless of processing (LPC or endo), gears of all sizes are shotpeened to instill compressive stresses into the hardened case of the tooth profile. Even high-pressure gas quenching (HPGQ) can leave tensile stresses in the carburized case that have a negative impact on gear performance. Shot peening for just a few minutes can increase the residual compressive stress levels to at least 700-800 MPa (101,500-116,000 psi).

Pits and Retorts vs. Batch Furnaces

Enter the "Green Energy Initiative." The proliferation of wind turbines has focused new attention on IGO due to the huge stresses encountered by those massive gear boxes positioned atop 300-foot towers. Jumping on the bandwagon of this renewable-energy effort, pit-furnace manufactures and their supporters claim that IGO can only be controlled in their retort-lined carburizing furnaces. Why pit furnaces? Because there was no other alternative for carburizing large gears and, for that matter, any large part.

Was the pit-furnace quality argument anything more than marketing propaganda? AFC-Holcroft planned an experiment

Fig. 1. Typical IGO

to compare three common steel grades and their reaction to three different endo-gas furnace environments. Following that first investigation, a cry for help was received from a captive transmission manufacturer struggling to reduce IGO in their single-row, four-zone pusher furnace.

IGO, Material and the Endo Atmosphere

It has been reported that steel chemistry has a direct effect on IGO development. To prove that claim, we exposed 8620, 9310 and 20MnCr5 to the identical carburizing atmosphere. Also, to investigate the effect of CO concentration on IGO, we altered the atmosphere as well. In all situations, we concerned ourselves with IGO depth only.

Manganese, chromium and silicon have been associated with contributing to the formation of IGO. Manganese is considered to be the most influential since it is present in all steels to fairly significant but varying amounts. Silicon and chromium also vary but may not be present at all. Silicon may exist as a trace element when not explicitly indicated.

It is pretty much accepted that a portion of the manganese existing in the grain boundary is there due to solid-state diffusion from the surrounding matrix, thereby depleting the hardenability of that immediate area around the IGO and exacerbating its effect. This phenomenon can be minimized by a higher-velocity quench.

To ensure an exact atmosphere composition for the tests, premixed bottled gases were used with the following compositions:

- 18.3% CO, 39% H_2, 40% N_2, 0.2% CO_2
- 14.3% CO, 54% H_2, 32% N_2, 0.2% CO_2
- Added methane to control carbon potential (CP)
- CP computed with three-gas analyzer

Figure 2 shows the test set up, which consisted of a 2-inch-diameter electrically heated 330 SS tube furnace with gas-mixing apparatus.

IGO Test Procedure

The following was used as the procedure for testing IGO.

- Heat tube to 1700°F (926°C)
- Purge the tube with the selected endo-gas mix
- Insert three steel samples
- Adjust the CP and carburize for selected time
- Dump quench samples into oil
- Remove samples, cut, mount, polish and read unetched IGO depth

Table 1 breaks down the relationship between environment, gas mix, temperature, time and IGO depth. Also indicated for comparison is the IGO obtained in a UBQ batch furnace processing grade 18 CrNiMo 7-6 gears carburized for 38 hours at 1725°F (940°C).

To investigate the furnace environment's effect on IGO, the gas inlet end of the 330 SS tube (Fig. 3, internal) was left unfilled to simulate a retort pit furnace filled with insulated firebrick (IFB) for a brick-lined furnace and ceramic fiber for a fiber-lined hot zone. In each case, the CO level was changed as was the temperature, carburizing time and CP.

The effect on IGO can be clearly seen, especially with the higher alloy 9310. It can be concluded, at least from this limited investigation, that IFB and ceramic fiber have no detrimental effect from the endo atmosphere and development of IGO. The higher CP, however, tends to lower IGO more significantly in 9310, and it has little or no impact on 8620 or 20MnCr5. Reducing the carburizing time agrees with other investigations stating that less time means less IGO. Drastically increasing the temperature by 150°F (66°C) slightly deepens IGO but not by an exaggerated or commensurate amount. It appears that IGO forms rapidly at first, and then the rate of increase slows.

A Real-World Problem

Not long ago, I visited a company that was experiencing excessive IGO (24µ) on 20MnCr5 gears when carburizing at 1700°F (926°C) in a brick-lined, gas-fired, single-row pusher furnace. The specifications were:

- Surface hardness: 58-63 HRC
- IGO (GBO) max.: 20µ max.
- ECD: 0.95-1.4 mm (pitch diameter)
- Retained austenite: < 30%
- No network carbide
- Grain size: ISO index 7

The four-zone, three-trays-per-zone furnace was operating on nitrogen/methanol set for a modified endo-equivalent mix

Table 1. IGO depth (microns) for steels carburized as shown

	Carburized for 24 hours @ 1700˚F (925˚C)				Carburized for 4.5 hours @ 1700˚F (925˚C)	Carburized for 7 hours @ 1850˚F (1010˚C)	Carburized for 38 hours @ 1725˚F (940˚C)
	CO 18.3%	CO 14.8%	CO 14.8%	CO 18.3%	CO 18.3%	CO 18.3%	CO 20.0%
	CP 0.82%	CP 1.16%	CP 1.16%	CP 1.04%	CP 1.05%	CP 1.05%	CP 1.18%
Sample environment	(Alloy[1])	(Alloy)	(IFB)	(Fiber)	(IFB)	(IFB)	(IFB) Flame metal
Steel							
20MnCr5	23*	20	22.9	25.4	15.3	17.2	
9310	18	12.7	12.7	15.2	7.6	20.3	
8620	25	25	24	22.8	10.2	27.9	
18CrNiMo7-8	→						20.0

*25.4 microns = .001", [1]Simulates the retort

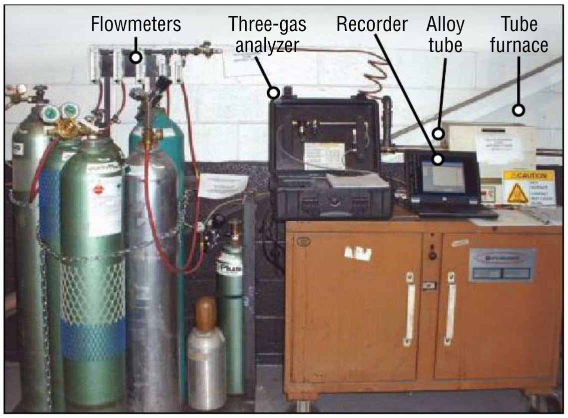


Fig. 2. Test setup

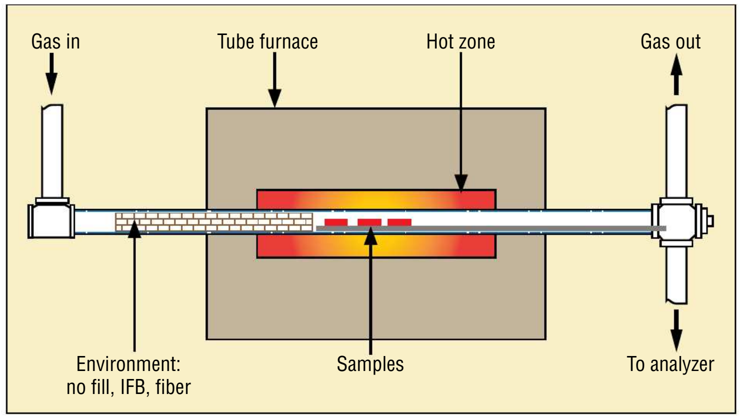


Fig. 3. Furnace schematic

and 55-minute push cycle with a 700°F (371°C) air preheat. Zones were initially set up as follows:

- Zone 1, heat to 1700°F (926°C), 2.75 hours
- Zone 2, carburize at 1700°F (926°C), 1.16% CP, 2.75 hours
- Zone 3, carburize at 1652°F (900°C), 0.95% CP, 2.75 hours
- Zone 4, diffuse at 1580°F (860°C), 0.85% CP, 2.75 hours
- Quench into 160°F (71°C) oil
- CP was verified by shim combustion analysis

Atmosphere Reactions

The only source of free oxygen in the endo atmosphere should be the dissociation of CO in contact with the steel surface. Carbon diffuses into the steel and oxygen is released. CO_2 can form via the reverse reaction with carbon at the steel surface (decarburization/equilibrium reactions). In addition, CO_2 can form when reacting with free oxygen and residual carbon (soot). Plus, free oxygen can form water vapor with hydrogen.

An effort was then made to reduce the time free oxygen existed in the atmosphere by maximizing the use of enriching gas (natural gas) and eliminating the need to add air to control the CP early in the process. Although some air was added in zone four, it was late in the process and exits quickly via the discharge vestibule effluent. The gas reactions in the endo atmosphere are continuously changing as the radiant-tube burners fire on/off. The load's surface carbon is getting ever richer, which changes the carbon demand, and the CP loop via on/off control adds natural gas. Because these reactions are constantly changing, the CP can be controlled to very accurate average values in very complex and dense loads. These reactions cannot be changed, but the time the gases are in contact with the parts can.

Increasing temperature has two beneficial effects. It can reduce IGO by limiting the steel's exposure to endo gas, and it can increase production. Increasing the carburizing temperature to 1750°F (954°C) reduced the push time from 55 minutes (11 hours in the furnace) to 37 minutes (7.4 hours in the furnace) – a 148% production increase. IGO is reduced from 24µ to 16µ while still maintaining all of the aforementioned specifications including grain size (Tables 2a & b).

The Magic of Shot Peening

Quenching to martensite no matter the method can, depending on the steel hardenability, subject the part's near-surface case to very high tensile stresses.

Most gears, even those subjected to

Table 2a. Test data

Original set up 55 min. push	Metallurgical Inspection: IGO <= 24 microns ECD @ pitch diam. = 0.99 @ 440 HV - ECD @ root diam. = 0.78 @ 550 HV			
Parameter	Zone 1	Zone 2, CP 0.80%	Zone 3, CP 0.80%	Zone 4, CP 0.75% [20 CFH air]
Temperature °C	910	909	890	850
Methanol (CFH)	160	160	160	160
Nitrogen (CFH)	250	150	150	250
Shim % CP		0.80	0.80	0.90

Table 2b. Test data

Final set up 37 min. push	Metallurgical Inspection: IGO <= 16 microns ECD @ pitch diam. = 1.04 @ 550 HV - ECD @ root diam. = 0.83 @ 550 HV			
Parameter	Zone 1	Zone 2, CP 1.40%	Zone 3, CP 1.00%	Zone 4, CP 0.75% [15 CFH air]
Temperature °C	954	954	926	860
Methanol (CFH)	240	240	100	0
Nitrogen (CFH)	160	160	300	400
Shim % CP		1.22	1.09	0.85

HPGQ, may require a final grind to bring the tooth profile into conformance followed by shot peening to induce compressive stress. Automotive gears all receive shot peening with or without a final grind. Shot peening provides several advantages in addition to creating compressive stresses. One reported advantage is that it provides a surface conducive to lubrication where microscopic roughness allows the lubricant to cling more readily to the gears. But the primary location gears see improvement is in the tooth root, where grinding is not done and where shot peening has its most pronounced effect with or without IGO.

I believe it is safe to say that in the rare circumstances where a gear has failed in the root it is not due to the presence of IGO but to a less-than-ideal quench exacerbated by a lean case depth. Investigations by others have linked non-martensitic transformation products (NMTP), such as bainite surrounding the IGO, to these failures. In discussions over the last few years with informed wind-turbine gear manufacturers, it has been stated that nowhere can it be reported that a gear has failed due to a root IGO issue.

Conclusion

Finally, closer attention paid to CP control; a more effective quench eliminating NMTP; steel chemistry control; low-side manganese; near eutectoid surface carbon; reduced carburizing time via higher carburizing temperature; and shot peening can essentially eliminate the negative effects of IGO. **IH**

For more information: Contact Jack Titus at AFC-Holcroft, 49610 Pontiac Trail, Wixom, MI 48393; tel: 248-668-4040; fax: 249-668-5571; e-mail: jtitus@afc-holcroft.com; web: www.afc-holcroft.com

EZ Lynks for Large Parts

The development of EZ Lynks™ by AFC-Holcroft was the direct result of witnessing wind turbine and other large gears being carburized and quenched using pit furnaces. The awkward manipulation via overhead crane and the fireball resulting from open-pit oil quenching made us realize there has to be a better way. The key to EZ Lynks' advantage is an enclosed quench tank and a rotating and multi-position transfer car. Any component can be removed with precise positioning for a free quench in water, oil or salt plus the possibility of press quenching, which is impossible from pit furnaces. Similar to our E-Z endo generator, which requires no retort removal head room, EZ Lynks requires no pit or additional head room due to the sideways-opening door and horizontal electrical or gas-fired radiant tubes.