Production Carbonitriding in LPG/CO$_2$ /NH$_3$ atmospheres
In Batch Integral Quench Furnaces

P.A.Balakrishnan & B.K.Venkatesh
(Techmat Enterprises (I) Pvt. Ltd.Chennai, India)

Pratap Ghorpade & P.R.Suresh
(Hightemp Furnaces Ltd
Bangalore, India

The FC-35 process originally introduced in Japan$^1$ otherwise known as “Fine Carburising® process” is widely used in a Production environment in Batch Sealed quench Furnaces for Carburising and Hardening applications. In this process, liquefied petroleum gas (LPG) and Carbon dioxide gas (CO$_2$) is used as a direct feed for producing carburising atmosphere in the furnace. The use of direct feed Hydrocarbon /Air$^2$ and LPG/CO$_2$ atmospheres is well documented. The FC35 atmosphere used with Ammonia (NH$_3$) is reasonably successful for Carbonitriding treatment of wrought steel parts also. This paper describes the Carbonitriding process in Sealed quench furnaces in FC35 atmospheres and highlights the flexibility of the process as an alternative to the nitrogen/ methanol atmosphere. The results of a variety of steel parts that were Carbonitried have been described also.

Introduction:

The FC-35 Process using Hydrocarbon (LPG)/Carbon dioxide (CO$_2$) atmosphere was developed primarily for cost and energy savings in comparison to the classical Endo gas or the alternative Nitrogen-Methanol atmosphere$^4$. The Nitrogen-methanol system became popular in India as a cheap substitute to Endogas atmosphere with the availability of low cost, high purity Nitrogen generated through on site PSA air separation and other similar equipments and also due to the free availability of Methanol. This also eliminated the need for high cost Endothermic generators and LPG bullet installation. However with the rising price of Methanol and also the strict Government restrictions in India in the handling and use of Methanol, many heat treaters are looking at alternatives to the once popular nitrogen-methanol atmosphere system. The FC-35 process developed and introduced in Japan and later successfully established in India$^4$ in Batch Sealed Quench Furnaces by M/s Hightemp Furnaces Ltd is a very attractive alternative to Endo gas or Nitrogen-Methanol atmosphere systems. Apart from cost and energy savings the other advantages of FC-35 process is in its flexibility, excellent metallurgical quality, shorter process times, and reduced furnace conditioning requirements.

Thanks to the Government of India’s liberalisation policy of industrial licensing, LPG is readily available in typical 19 & 33 Kg. Cylinders. The cost of installation of LPG/ Carbon dioxide cylinder banks with manifolds is very low compared to a large LPG bullet installation or Nitrogen PSA generators and Methanol pumping and storage systems. Ammonia (NH$_3$) required for the Carbonitriding process is sourced from typical 50 kg cylinders.

CARBONITRIDING IN FC-35 ATMOSPHERE

The carbon potential of the in-situ generated atmosphere in the FC- 35 process is monitored and automatically controlled in the batch furnaces using oxygen probes. The probe EMF (millivolt) produced is
used as the input signal to the Carbon Potential controller to control additions of the carburising gas (LPG) through a motorized actuator and a port valve into the furnace. Ammonia and CO₂ flows are maintained constant throughout the process during Carbonitriding in the FC35 process. Carbonitriding is a thermochemical treatment usually conducted at temperatures in the range of 800-900 °C. As during carburising, when a controlled level of carbon is introduced at the surface and allowed to diffuse to the required depth; in Carbonitriding, nitrogen is also imparted, along with the carbon, to improve case hardenability. In gaseous Carbonitriding, both carbon (from additions of LPG) and nitrogen (from additions of NH₃) diffuse into the surface of the steel, simultaneously and change the chemical composition of the surface of the part. Subsequent fast cooling by quenching in oil in the sealed quench furnace produces a hard case combined with a soft/tough core. The quenching is normally followed by a low temperature tempering/stress relieving treatment. The FC 35 process Carbonitriding uses a carburising atmosphere produced in-situ from a mixture of LPG (composed of approximately 60-70 % Butane and 30-40 % Propane, available in India) and commercial quality CO₂ with addition of NH₃. The transfer of carbon from LPG to the steel surface takes place via the reaction:

$$2CO \rightarrow C + CO_2$$

The nitrogen activity is produced from NH₃ as expressed by the following reaction:

$$2NH_3 \rightarrow 2N + 3H_2$$

**CARBONITRIDING PROCESS USING FC 35 ATMOSPHERE**

A typical Carbonitriding process cycle using the FC35 atmosphere would be as follows (Fig 1):

- Prewash
- Condition/Season furnace atmosphere at 880°C (Carbon Potential 0.85%), using only LPG/CO₂
- Load furnace and recover to set Carbonitriding temperature as desired by heat treat specifications and chemistry of part (Typically 800-880°C) - carbon potential is controlled automatically during recovery in relation to set temperature.
- Carbonitride at set temperature and set carbon potential (CP) for required period. Varying the LPG flow automatically by means of a motorised valve automatically controls the carbon potential. CO₂ flow volume is set at a constant value. NH₃ volume is also set at a constant value.
- Quench in oil after set time period has elapsed.
- Post wash and temper

A typical process cycle would be as follows:

1. Prewash
2. Carbonitride and Quench in a Sealed Quench Furnace
3. Post wash and Temper

**PROCESS EVALUATION**

Evaluation of the FC 35 process for single stage Carbonitriding was carried out using a series of nine test pieces in a charge (placed at eight corner positions and the ninth piece at the centre of the charge basket) in order to assess the uniformity of Carbonitriding treatment results. The test pieces were examined for case depth quality, hardness traverse profile and surface hardness.

**SAMPLE LOCATION**
HARDNESS TRAVERSE PROFILE AND CASE DEPTH EVALUATION

Figure 2 shows the Hardness traverse profile tests, and Figure 3 shows the Effective Case Depth values measured on nine samples in a typical full load batch of an IS 513 Type “D” grade steel. The component is a typical machined part.

Figure 2:
The variation in the effective case depth in the nine samples was in the range of 0.17 to 0.20 mm.

Figure 3:
The effective case depth for this part is defined as a depth of the part surface layer with a minimum hardness of 50 HRC (513 HV1)

Material Specifications:

**IS 513 Type “D”**
- Carbon = 0.12 % – 0.15 %
- Silicon = 0.10 %
- Sulphur < 0.04 %
- Phosphorous < 0.04 %

CASE DEPTH AND SURFACE HARDNESS IN PRODUCTION BATCHES

The following results are compiled from carbonitrided production batches on a typical fine blanked toothed engine component. The uniformity in surface hardness (measured on a Hardness Rockwell A Scale) and the case depth variation, are within acceptable limits as seen in Figures 4 and 5.

The Surface hardness specified on the toothed area is 75-83 HRA.

The Case Depth is specified as the depth, at which the hardness measured on an HV0.3kg scale is 50 HV more than the core hardness. The material is IS 513 Type “D”. The specification requires a Case Depth of 0.50 to 0.80 mm.

These results were tabulated from 25 continuous production batches; to demonstrate the stability of the FC 35 process when used for Production Carbonitriding, in a batch type Sealed Quench Furnace.

It can be seen that Carbonitriding using the FC35 process is an effective and stable process.

Figure 4:
Batch-to-Batch Surface Hardness results for a Fine Blanked Toothed Engine Component, Carbonitrided in FC35 atmosphere
Material: IS 513 Type “D”
Figure 5: Batch-to-Batch Case Depth results for a Fine Blanked Toothed Engine Component, Carbonitrided in FC35 atmosphere
Material: IS 513 Type “D”

COMPARISON OF RESULTS WITH NITROGEN- METHANOL AND FC35 ATMOSPHERES

Carbonitriding in conventional Nitrogen –Methanol and also in FC35 atmosphere were conducted on a typical full load of Fine Blanked Two wheeler sprockets made out of 15 Cr3 steel, for a comparative study.

Component surface hardness and Hardness traverse profiles on nine samples tested from both these loads are shown in Figures 6 and 7.
The specifications call for a Surface Hardness of 77-83 HRA, and a Case Depth of 0.50-0.70 mm (HV0.3kg/550)

Figure 6:
Hardness Traverse Profile comparison for 15Cr3 material sprockets in FC35 and Nitrogen-Methanol atmospheres

It can be clearly seen that the results obtained in the FC35 process is comparable to that treated in the Nitrogen-Methanol atmosphere, and were considered to be of an acceptable quality standard

MICRO STRUCTURE OF A PART CARBONITRIDED IN FC-35 ATMOSPHERE:

Figure 8:
Case Microstructure Consists of Fine Tempered Martensite with approximately 10 % Retained Austenite

Mag: 400X
CONCLUSION:

(1) FC35 process using Ammonia is a stable process for Carbonitriding, and the results are comparable to Carbonitriding in Nitrogen Methanol atmospheres with Ammonia.

(2) With the proven cost advantages of the FC 35 process Carbonitriding in FC-35 atmospheres gives an added advantage and makes it an attractive and viable for production heat treatment.

References


2) Hewitt W., ‘Users View of Carburising with Direct Feed Hydrocarbon/Air Atmospheres (Supercarb)’ – Heat Treatment of Metals Vol. 27 2000.2
