

# Sub-zero Treatments

Sub-zero treatments are simply those treatments where components are cooled below room temperature. There can be many reasons to do this, but the main ones are to remove retained austenite from quenched components or tools, to increase the wear resistance of tools or to stabilise the component.

### Retained austenite

Austenite is the soft phase in steels that is stable at high temperature. On rapid cooling or quenching this phase is converted to hard martensite. However, in many tool steels the temperature needed to get conversion to a fully martensitic structure is well below room temperature usually in the range -70 to -120°C. It is possible to convert the austenite "retained" in the structure to martensite by multiple tempering, but it is more efficient to cool the tool to say -120°C using liquid nitrogen then do a single temper. An example of the effect on D2 is shown in Figure 1.

Carburised steel components can suffer from the same problem if they are carburised to a higher than optimum carbon level. They can also be cold treated in the same way to increase the case hardness and avert grinding cracks. However, if the carbon level is too high the temperature needed may be below absolute zero so complete conversion is not possible. A typical example is shown in Figure 2.

## 16 Retained Austenite (%) 12 120°C/25 m 10 -150°C/25 m 8 ■ - -150°C/24 h 6 2 1 temper 2 tempers 3 tempers As After quenched cryogenic treatment

Figure 1. The effect of cold treatment on retained austenite in D2

#### Wear Improvement

When martensite is subjected to deep cold around the boiling point of liquid nitrogen (-196°C) for extended periods, dislocations agglomerate into nucleation sites. When the steel is subsequently tempered the fine η-carbides precipitate at these sites. It is generally acknowledged that an improvement in tool wear results from the formation of large numbers of nanoscale coherent η-carbides. When the processing is carried out correctly, some significant improvements have been reported - up to ten times the life for D2 forging dies.

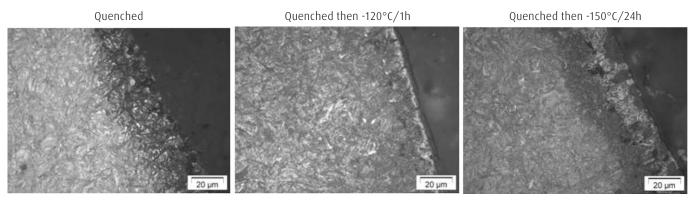


Figure 2. A comparison of the case microstructure of 21NiCrMo2 after various cold treatments (the retained austenite is the white phase between martensite laths)

#### Stabilisation

Some high precision steel components, such as bearings, gun barrels (Figure 2) and rolling mill rolls, are hardened before being put into service and are then exposed to high stresses and temperatures in use. Under these conditions, metastable phases formed during hardening can transform and cause distortion. The distortion involved is usually very small, but can be critical to the performance of these components. In some cases the distortion can be larger and is itself caused by cold - as one exporter of circular saw blades discovered when blades sent by air in an unheated cargo plane arrived too warped to be used.

One way to stabilise these components is to deep cold treat them before they are put into service. Deep cold treatment at temperatures between -70°C and -150°C transforms any retained austenite present in the microstructure to martensite. Tempering at a suitable temperature for the steel produces a fully stable structure. The component may then be machined to its final shape and will not distort during use.



Figure 3. Deep frozen gun barrels (Photo courtesy of Cryoplus Inc.)

# Equipment for cryogenic processing

Processors can use liquid nitrogen effectively to achieve the temperatures necessary for deep cold treatment and to get quick cool down rates. One of the most common techniques is to use a spray header system with atomizing nozzles that convert the liquid nitrogen to very cold gas, as the liquid nitrogen flashes to a vapour and warms up. Only cold gas and not fine droplets of liquid should come in contact with the surface of the part being cooled to avoid "spot martensite" formation. It is possible to control the temperature by controlling the nitrogen flow. Direct cooling is the most efficient way to obtain low cryogenic temperatures for controlled processing.

Cryogenic chambers come in a variety of sizes and configurations. The toploading Cryogenic Box Freezer, CRYOFLEXTM-CBF in Figure 4 offers an economical solution for tool makers and users. By using liquid nitrogen as the cooling medium, the chamber is suitable for cryogenic treatment and deep cold processing. The chamber can be loaded manually or by a hoist or overhead crane. The interior is made of stainless steel, as are all piping and components that are exposed to the liquid or cold nitrogen gas.



Figure 4. A top loading cryogenic treatment box

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