Delta ferrite content of wetted materials

1 Introduction

Austenitic stainless steels, such as 1.4435 or 1.4404, are used regularly in the pharmaceutical industry as they are resistant to the usual process and cleaning media.

Depending on the alloy components and the heat treatment, austenitic stainless steels may contain a proportion of delta ferrite. This can have a negative influence on corrosion resistance, which is why the proportion of delta ferrite should be limited.

2 Formation of delta ferrite

During rapid cooling of austenitic alloys, the austenitic structure can be partially transformed into a ferritic structure. This cooling can occur during the production of the raw material or through a subsequent processing step. Here, welds are particularly important.

Austenite can partially convert into so-called deformation-induced martensite in the case of mechanical loads (deformation, machining). However, a transformation into delta ferrite only occurs when the workpiece has heated up significantly during mechanical processing, e.g. when drilling without cooling lubricant.

2.1 Influence of the alloy components

The tendency towards the formation of delta ferrite upon cooling is crucially dependent on the alloy composition. A distinction is made between ferrite formers (chromium, molybdenum, etc.) and austenite formers (nickel, cobalt, manganese, etc.).

The material standards for stainless steel only define spans for the alloy components. Different material batches with the same material number can therefore show a different tendency towards delta ferrite.

Over time, various methods have been developed to predict the formation of ferrite after welding. The WRC-1992 diagram [WRC-1992] (a further development of the well-known Schaeffler diagram) is currently considered the best method for this purpose.

When using this method, the so-called chromium and nickel equivalents are calculated from the alloy components of the stainless steel. These values can then be used to determine the maximum expected proportion of delta ferrite after welding with the WRC-1992 diagram.

3 Determination of the delta ferrite content

Delta ferrite and austenite differ only in the crystal structure but not in the chemical composition. This limits the analytical possibilities. Destructive measurements are accurate, but cannot be performed on the final product. A non-destructive measurement is subject to uncertainties.

3.1 Metallographic method

The safest method is a metallographic examination. A sample is ground and etched and then evaluated under an optical microscope. Due to the pre-treatment, the delta ferrite differs from the surrounding austenite because of its lower brightness.

3.2 Magnetic inductive method

The non-destructive, magnetic inductive method for determining the delta ferrite content uses the fact that delta ferrite is magnetisable, but austenite is not [FISCHER].

The disadvantage of this method is that all magnetisable components are identified, not just the delta ferrite. This includes in particular the above-mentioned deformation-induced martensite, which is also magnetisable.

It is also important to note that measurements on thin-walled parts, bends and edges are prone to errors. Furthermore, the spatial resolution of the measurement is limited. Conventional measuring devices therefore require a minimum volume of a 2-3 mm edge length according to the magnetic inductive method. A mathematical correction of these effects is possible in some instances, but leads to a further loss of measurement accuracy.

4 Normative requirements

In relevant standards, which provide information on the determination of the delta ferrite content (e.g. [DIN11865], [DIN11866], [BN2]), all magnetisable structural components are combined for the limit value and a magnetic inductive test is proposed.

5 Application on LABOM diaphragm seals

5.1 Wetted surfaces

The by far largest part of the wetted surface of a diaphragm seal is usually the diaphragm. Part of the base body may also be wetted depending on the type of diaphragm seal. With few exceptions, the diaphragm and the base body are connected by means of laser welding with a seam width of a few tenths of a millimetre. No further heat treatment takes place.

5.2 Assessment

The diaphragm is only deformed. As these are very thin foils, the degree of deformation is low. The delta ferrite content of the embossed diaphragm therefore corresponds to the content of the raw material.

The machining of the base body is generally carried out with cooling lubricant. Due to the required surface quality, the final machining step is also performed with low feed and chip removal, so the formation of delta ferrite can also be ruled out during production of the base body.

Joining by means of laser welding is a possible reason for the formation of delta ferrite. However, the heat-affected zone is very small in laser welding so only a very small proportion of the wetted surface is at risk.

It is mainly the diaphragm that melts during the welding process. This is one of the reasons why 1.4435 is used as a diaphragm material as it tends to form less delta ferrite due to the higher nickel content compared to 1.4404. In case of special requirements regarding the delta ferrite content, the alloy composition per the raw material certificate and the WRC-1992 diagram is used to identify a suitable raw material, as proposed in [ASME BPE].

5.3 Verification of the delta ferrite content

As described in chapter 5.2, the raw material certificates can be used for the diaphragm and the main body as verification of the delta ferrite content. Verification for the weld seam is difficult due to the limited spatial expansion (see chapter 3.2) and of questionable value due to the measurement inaccuracies.

Despite this, a certificate related to a magnetic inductive measurement of the delta ferrite content can of course be issued on request.

Sources

[ASME BPE] ASME Bioprocessing Equipment 2019

[BN2] Basel Standard 2 issued by the Basel Chemical Industry (BCI)

[DIN11865] DIN 11865:2012-02
Stainless steels fittings for aseptic, chemical and pharmaceutical industry - Tees, elbows and reducers for welding on

[DIN11866] DIN 11866:2016-11
Stainless steel components for aseptic applications in the chemical and pharmaceutical industry - Tubes

[FISCHER] https://www.helmut-fischer.com/techniques/ferrite-content

[WRC-1992] D. J. Kotecki and T. A. Siewert,
"WRC-1992 Constitution Diagram for Stainless Steel Weld Metals: A Modification of the WRC-1988 Diagram", Welding Journal 71(5), p. 171-s, 1992.