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REMOTE VISUAL EXAMINATION OF STAINLESS PIPELINES FOR PHARMACEUTICAL INDUSTRY

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ABSTRACT

For the construction and maintenance of stainless pipelines the remote visual examination is employed. The weld roots and the pipe-wall inside are inspected to detect possible defects that could result in corrosion and bio-chemical processes. Such defects are mechanical and thermal damages to the oxide layer and defects in the weld roots. The remote visual examination is mandatory with welding and mounting of pipework systems. It is also used in regular inspections of the inner surfaces and welds during pipe operation in order to detect locations of potential corrosion and nests of micro-organisms.
The welded pipelines made of stainless steel should fulfil relevant general and sanitary requirements. There should be neither undercuts nor unevenness at the root side since colonies of micro-organisms may occur at such areas. The oxide layer protecting a metal against corrosion should not become dark-coloured close to the weld due to a thermal influence.
The paper treats sanitary requirements concerning stainless pipelines used in pharmaceutical factories. Weak points at the weld root side are shown. The relationship between colouring of the over-heated area and corrosion resistance of the welded joint is indicated. The importance of the remote visual examination and the assurance of weld quality in the production of equipment for the pharmaceutical and food-processing industries is underlined).

***Keywords**:* Welding, Pharmaceutical equipment, Stainless steel pipelines, Sanitary requirements, Pitting corrosion

1. Introduction

In the manufacture of pharmaceutical products stricter and stricter standards specifying permissible contents of impurities in final products are being implemented. It is known that rough and damaged inner surfaces of manufacturing devices are an excellent environment for various biochemical and corrosion processes polluting products. Production lines should, therefore, be carefully designed and constructed. Particular attention should be paid to welded joints in piping for processing water of different types such as purified water (PW), water for injections (WFI), clean steam (CS), and purified steam (PS).
The water quality, that is the allowable content of impurities in water, is specified in various pharmaceutical standards and guidelines of the associations of pharmaceutical firms. In the manufacture microbiological pollution cannot be avoided completely but it can be kept under control by keeping the contents of micro-organisms and metal ions within admissible limits. It

should always be taken care, that in design, construction and maintenance of the piping, there are as few as possible locations where biochemical processes may start. Pipe systems linking production lines to the water-preparation facilities should be designed as closed loops. The water in the system shall constantly circulate. The unconsumed water shall return to the facility where the micro-organisms appearing will be eliminated by ionization and ultraviolet irradiation.
In the pharmaceutical industry special quality-assurance systems are being introduced. They include special requirements regarding the quality of welded joints called sanitary requirements. Standards and guidelines to specify such requirements are under preparation [1]. For the moment the recommendations of various pharmaceutical associations and client requirements are being implemented. The American Welding Society (AWS) prepared a specification for welding of tube and pipe systems for sanitary applications [2]. There is also an ASME standard on welding and control of pipelines for sanitary purposes [3].

2. Quality of welded joints

The tubing dedicated to processing water in pharmaceutical applications should not only show sufficient strength and tightness but also meet sanitary requirements, which means that welded joints produced should not contaminate pharmaceutical products. Special attention should be paid to the root side of welds. The weld and the heat-affected zone should not contaminate products by metal ions and micro-organisms.
The tubing systems for processing water in the pharmaceutical applications are usually made from high-quality austenitic stainless steel such as X2CrNiMo 18-14-3 (316 L). Tubes shall be welded longitudinally. The inside surface shall be ground and polished. The most suitable are the electro-polished tubes of which roughness (Ra) does not exceed 250 nm. Consequently, at the inner surface of the tube a thin compact oxide layer protecting metal against corrosion will form.
The thin compact oxide layer of a few nanometres in thickness and containing a sufficient content of chromium oxides will protect stainless steel against corrosion. According to literature data corrosion resistance can be promoted by as much as 10% Cr in the oxide layer [4]. In more aggressive corrosion conditions, however, the oxide layer should contain at least 13% Cr. The composition and structure of the alloy below the oxide layer and their relation are certainly important as well. The corrosion resistance of stainless steel is affected not only by its chemical composition but also by the physical properties of the oxide layer, which should be thin, compact, and without cracks. The metal will thus be well protected by a glasslike oxide deposit. Such a deposit is well-suited to pharmaceutical purposes since nests of micro-organisms cannot stick to it.
The chemical composition of the oxide layer is difficult to determine. In the literature different data may be found [4, 5]. A base of the insoluble layer consists of hydrated iron-chromium mixture oxides. An important role is played also by other components that may occur in the oxide film such as molybdenum, manganese, and nitrogen. The Auger electron spectrometry (AES) was employed to analyse the distinctly tinted oxide layers at X2CrNiMo 18-14-3 steel. It was found that in all the cases the chemical composition was similar. The oxide layer to a depth of 20 nm contained 60 at. % of oxygen, 20 at. % of chromium, 10 at. % of iron, and 4 at. % pf molybdenum. In the depth exceeding 40 nm there was no more oxygen and the iron content increased from 10 % to 60 %.
No matter how well performed a welding procedure is, it cannot ensure the same high quality of the oxide protection of the weld root as it can be ensured by grinding, polishing and passivation of the treated inside surface of the tube. This is due to various welding defects, oxidation of the zone next to the weld, a rough, cast surface of the root as well as chemical and structural changes occurring in the heat-affected zone. During welding oxidation will occur and the protective film will become thicker. As the zone next to the weld heats, the oxide-layer thickness will increase up to 100 times. Consequently, the layer will crack and get a spongy structure. The oxidation

process is going on at a temperature above 300 °C. The thickness of the oxide layer next to the weld depends on the temperature, heating time, and oxygen concentration in the shielding gas.

In the weld and the heat-affected zone structural and chemical changes will occur. The chromium dissolved in the matrix will form carbides in the vicinity of which the alloy will be locally depleted of chromium. Because of surface defects such as slag residues, poor weld ends and smaller undercuts, the protective layer will become weaker at some locations.

Corrosion processes will often start just next to the weld. There the oxide film is the weakest. In pharmaceutical devices the system contains pure water. It is a laic belief that such water does not initiate corrosion. According to the literature a break-through of the oxide layer is produced by chlorine ions, which, however, are not found in the processing water. A high content of dissolved oxygen produces the formation of hexavalent chromium ions, which are water-soluble. Thus a local failure of the oxide layer occurs where the oxide layer is the weakest. Pitting corrosion will initiate as well. Pits will be invaded by micro-organisms forming large nests from which they will migrate to water. Because of the chemical processes in water, metal ions will be transmitted to water (Fig. 1).

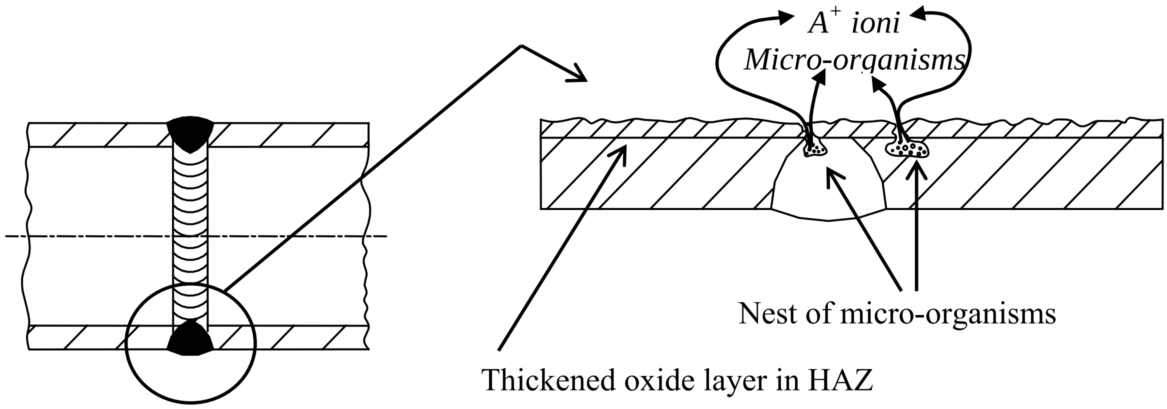


Fig. 1: Pitting corrosion next to weld.

An adequate welding procedure, a good root protection, and quality control and assurance will reduce the undesired welding effects to an acceptable degree.

Sanitary requirements are applied to the manufacture of pharmaceutical equipment. Recently they have been increasingly applied also to the food-processing industry. They specify how welded joints should be made not to contaminate products. It is not acceptable that the heated zone next to the weld be tinted dark. The root side of the weld should show neither surface defects nor bonding defects

3. Welding

In the pharmaceutical industry the automatic orbital TIG welding became established to secure high quality and reproducibility. It is used to manufacture tube systems supplying processing water to production lines. A program unit permitting to vary welding parameters continuously or step by step makes it possible to weld in all positions. The device is equipped with a microprocessor storing the programmes for welding of the entire range of different diameters and thicknesses. Welding is carried out in a chamber filled with a shielding gas.

The root side shall be additionally protected by a backing gas supplied through the tube. The shielding gas used is argon 4.8 containing 99.998 % Ar, and a mixture with an addition of 10% H_2.

Weld edges shall be prepared by a special device so that both tube ends fit well. The tubes shall be manually tacked.

Welding shall be carried out taking care that the material next to the weld heats as little as possible and cools as fast as possible. Oxygen, however, cannot be eliminated no matter how well the root is protected. According to literature data, the system should be purged as long as the shielding gas does not contain more than 40 ppm O_2 [5]. The shielding gas serves as a backing. The pressure of the shielding gas at the weld root should be somewhat higher than at the weld face. The correct ratio of the pressures at the two weld sides helps in shaping the weld and keeping the weld pool in out-of-position welding.
The oxide-layer thickness cannot be measured directly. Its colour, however, permits to approximately determine its thickness and corrosion resistance. Tinting at the root side is also a criterion for the assessment of the weld acceptability. Up to a thickness of 20 nm the oxide shows a metallic shine. An increased thickness results in changes of heat tints ranging from yellow, brown, blue to black ones (Fig. 2). A yellow tint is admissible at the root side, but at the weld face a blue tint is admissible too.

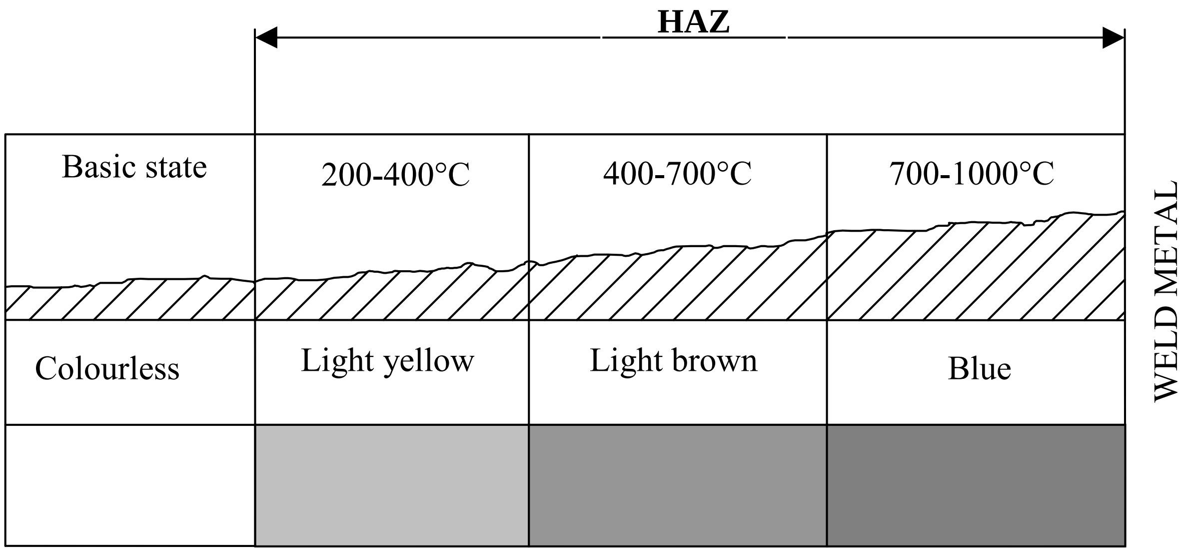
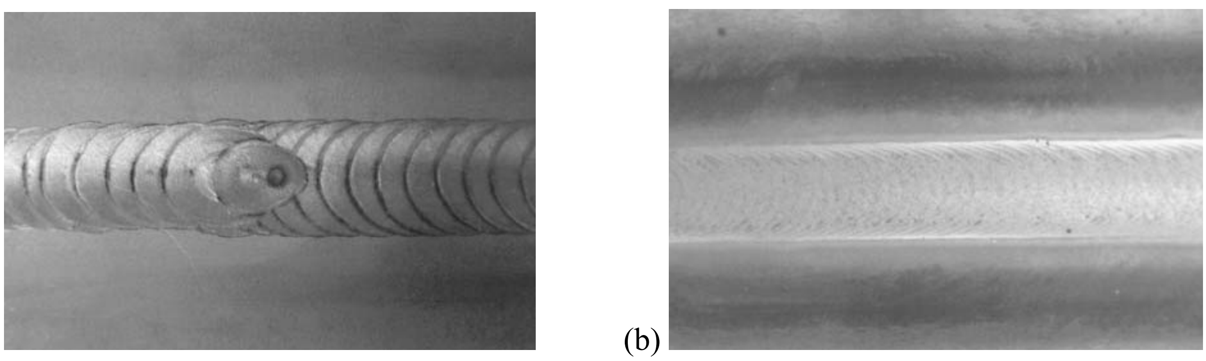


Fig. 2: Thickened oxide film in HAZ gets tinted.

The harmful effect of oxygen in the shielding gas shall be diminished by an addition of hydrogen that will bond with oxygen to make water steam at a temperature above 500 °C; therefore, in practical applications argon 4.8 or argon 5.0 with the addition of 10% H_2 are mostly used.
After welding, leaching and rinsing of the weld (passivation) are recommended [6-8]. Various nitrous-acid and phosphoric-acid based preparations are commercially available for this purpose. Passivation produces the iron-oxide extraction from the oxide layer. Consequently, the Cr content will increase.



(a) (b)

Fig. 3: Appearance of weld root (a) and weld face (b) produced by orbital welding of tube.

Figure 3a shows the root side of the weld. A final pit, which may be a potential spot for the initiation of corrosion processes, is visible. The heat-affected zone is slightly tinted, which indicates that the root shielding was efficient enough.
Figure 3b shows the weld face. The heat-affected zone is strongly dark-blue-tinted. Considering that the weld face is not in contact with the medium, tinting of the heat-affected zone is acceptable.

4. Quality assurance and control of welds in the production of pharmaceutical equipment

The top quality of welded joints required for pharmaceutical tubing cannot be accomplished without adequate quality control and quality assurance.
Qualified and certified welders are required for welding work. They should master not only orbital welding but also manual welding of stainless steel tubes. For all tube diameters, a manufacturer should have approved welding procedures and meet sanitary requirements.
The manufacturer should employ qualified personnel for co-ordination and control of welding work knowing well the pharmaceutical equipment. He should also meet all other quality requirements for welding work [9].
Before starting his daily work, each welder should weld control test pieces for all the diameters to be welded that particular day. These test pieces shall be stored and serve as reference specimens for the subsequent assessment of weld quality at the facility.
Welding of tubes for the pharmaceutical industry shall be tested by remote visual inspection (RVI). The camera used for the RVI gives a good view of the weld root and the tinted area along the weld. Modern devices make it possible to reach welds with a video-camera within 12 m from the inlet opening. Only tubes having a diameter of 8 mm and more can be inspected in this way. The root side shall be inspected from various viewing angles. The image obtained can be enlarged and dimensions of the defect detected can be measured. The images shall be stored on a video-cassette. An up-to-date videoscope is shown in Fig. 4.

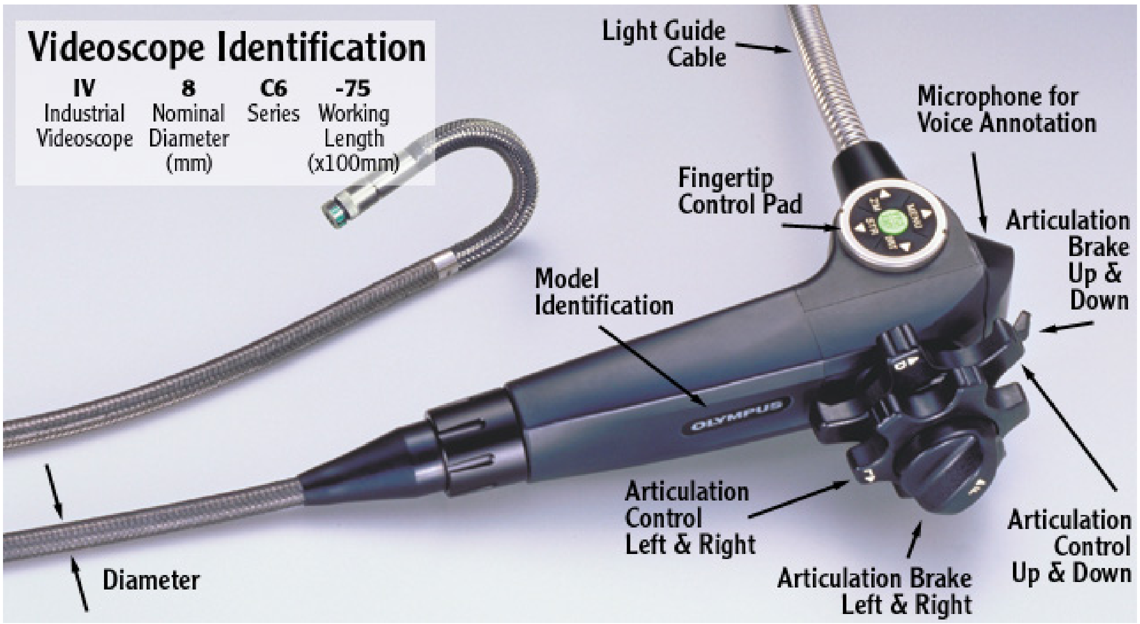


Fig. 4: Videoscope manufactured by Olympus.

At the end of a flexible tube there is a video-camera providing images to the display. The spot examined should be well-lighted. The device has a strong light source. The light is conducted

along optical fibres to the spot examined. A two-dimensional image obtained is slightly distorted.
The camera can provide a front view or a side view of the weld. Figure 5 shows respective views of the weld in the direction of the tube axis and in the direction perpendicular to the tube axis.

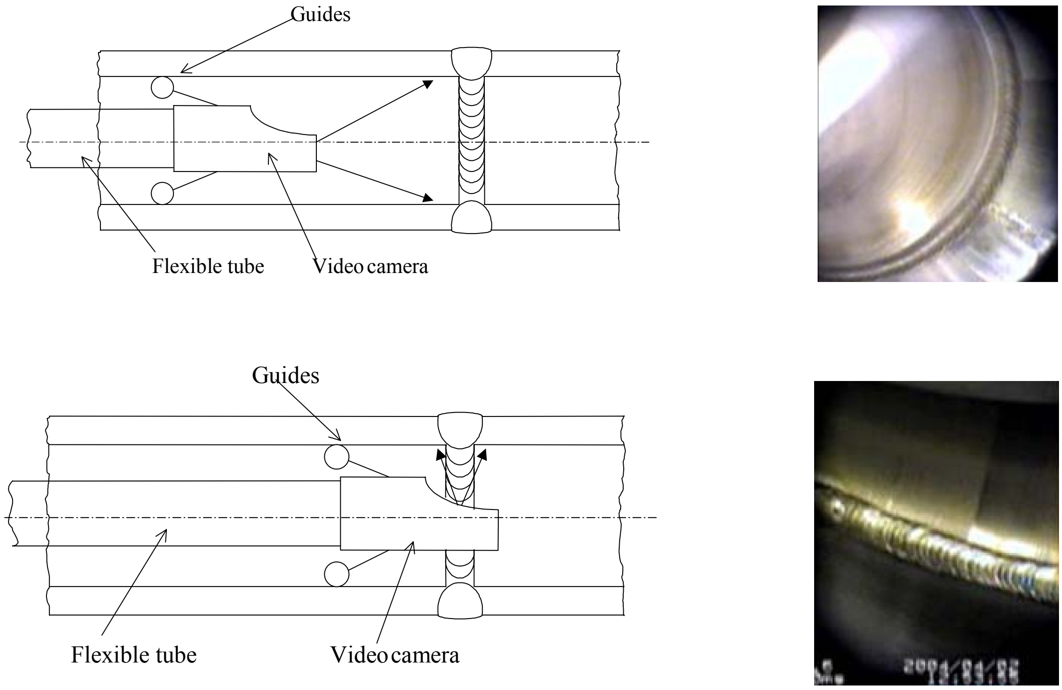


Fig. 5: Views of weld root in direction of tube axis and in direction perpendicular to tube axis.

The remote visual testing (RVT) is employed to detect various defects at the root such as lack-of penetration, misalignment, undercuts, an excessive root, slag residues, and poor weld ends.
The heat tints in the heated zone of the weld indicate whether the root side was adequately shielded.
Colour charts were elaborated for some steel types frequently used in pharmaceutical applications [10]. They show typical cases of tinting of the heated zone appearing with different oxygen concentrations in the shielding gases. When the oxygen concentration is higher than 15 ppm, the tints vary from yellow, green, blue to grey, and black. The said colour charts can be of great help in the determination of the acceptance level as well as in the control.
The visual examination shall be applied also to the weld face. Any shape defects at the weld face will show also in the weld root. The suspicious locations should be carefully examined also from the inside. The acceptance criteria for the weld face, however, are less stringent than for the weld root. The blue tint is admissible whereas the grey and black tints are not admissible.

5. In-operation remote visual inspections

In the production of medicines the content of ions and micro-organisms in processing water should be constantly monitored. If the content of impurities should increase, this means that in the processing devices used corrosion and biochemical processes have initiated.
On the occasion of maintenance work scheduled, the inside surfaces of process reactors and transport means shall be carefully examined with remote visual testing. Special attention should be paid to welds at tubing.
How the orbital welds of high quality actually behave can be established when the welds are removed from operation during maintenance. The welds being removed now in the

pharmaceutical industry are, as a rule, in poor condition. When tubes removed today were constructed, the quality requirements were not so stringent as they are today. It is assumed that the root side was poorly shielded; therefore, the occurrence of pitting corrosion should not come as a surprise [11-14].

Pits at the weld end appeared in some cases. At the tube inside a small patch of slag at the weld end was found to be a weak point for the occurrence of pitting corrosion. Some pitting corrosion was noticed also at the remaining part of the root, and some more at the weld edge [15]. Pits occurred also at the outside boundary of the heat-affected zone (HAZ). Corrosion appearance could be expected mainly next to the weld. At this location oxidation is expected to be the strongest because of elevated temperatures. The majority of pits, however, occurred at the edge of the blue zone where the tube does not heat above 350 °C. Another weak spot, on which nothing is reported in the literature, are the longitudinal weld and weld intersections of the longitudinal and transverse welds.

6. Conclusions

Based on the investigations conducted the following conclusions may be drawn:

- In the manufacture and maintenance of the equipment employed in the pharmaceutical industry sanitary requirements for the quality of welded joints should be fulfilled.
- Welded joints should be produced so that they may not contaminate products by metal ions and micro-organisms.
- The root shielding should be so efficient that the oxide layer in the heated zone next to the weld may not get thicker.
- The colour of the heated zone depends on the oxide-layer thickness. Light tints are admissible whereas blue and grey tints are inadmissible.
- To ensure sanitary soundness of the pipelines, all the welds should be subjected to remote visual inspection.

Remote visual testing is applied to construction of pipelines and maintenance of pipelines for processing water in the pharmaceutical industry.

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