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Faculty of Mechanical Engineering

The Effect of Welding Processon the Fatigue Life of Joints made by DiffusionWelding

Master thesis

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DIPLOMA THESIS ASSIGNMENT

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Rules for elaboration:

1. To get acquainted with the principles and fundamentals of the diffusion welding.
2. To carry out literature review concerning the principle of diffusion and possibilities of diffusion welding.
3. To carry out literature review concerning the low-cycle and high-cycle fatigue testing of basic materials and weld joints.
4. To get acquainted with material properties of steel 10GN2MFA.
5. To become familiar with possibilities and operation of thermos-mechanical simulator Gleeble 3500.
6. To propose and perform experimental program for diffusion welding.
7. To perform fatigue testing of the diffusion-bonded joints.
8. To evaluate experiments and make conclusions.

Scope of graphic works: **tables, graphs**

Scope of work report
(scope of dissertation): **approx. 50 p**

Form of dissertation elaboration: **printed/electronical**

List of specialized literature:

[1] **Kazakov, N.F. 1985.** *Diffusion Bonding of Materials.* **Moscow : Mashinostroenie Publishers, 1985. 0-08-032550-5.**
[2] **Kearns, W. H. 1980.** *Welding Handbook: Resistance and Solid-State Welding and Other Joining Processes.* **United Kindom : American Welding Society, 1980. ISBN 978-1-349-04961-5.**
[3] **Gleeble User Training 2012,** *Gleeble systems and application*, **DSI: 120419.**
[4] **AWS Welding Handbook:** *Welding Science and Technology*. **9th Ed., Vol.1, 2001.**

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Liberec, dated: 1 November 2017

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Abstract

To accomplish a specific task just like any other technology-oriented, the 10GN2MFA steel went for diffusion welding and the welded steel has gone through the fatigue mechanical test. Diffusion welding method used for improving joint interference, especially for fatigue strength, through atomic bonding along bainitic and pearlite microstructures. However, the purpose of the master thesis is to characteristics fatigue behaviour of 10GN2MFA basic and 10GN2MFA welded steel with the constant parameter. The heating and cooling rate of welding, chemical composition, and hardness highly influenced fatigue mechanical strength. The S-N diagram revealed after diffusion weld the 10GN2MFA steel reduced nearly 24% of fatigue strength. On other hands 400 MPa the welded steel fatigue limit could not be obtained even at 10^5 stress cycles. This test highly influenced towards stress amplitude, where fracture happened in the joint area above endurance limit. These results were compared to load to load groups with the same frequency of fatigue cycles. The thesis all main points were discussed in conclusion of this work.

Keywords

Diffusion Welding; Gleeble; 10GN2MFA Steel; Hardness; Microstructure; Fatigue

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Contents

List of tables, graphs, and images

Table List

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List of abbreviations and symbols used

Symbol	Units	Name
A_g	[%]	Elongation
C_A	[mol.m^{-3}]	Concentration of element
D	[m^2.s^{-1}]	Diffusion Coefficient
D_0	[m^2.s^{-1}]	Pre expontial factor
R	[-]	Gas constant
X	[m]	The Direction of Diffusion
T	[°C]	Temperature
Q	[J]	Amount of heat
I_{rms}	[A]	Value of current
R_s	[W]	Rate of heating
t	[S]	Time
E	[V]	Electro motive force
R	[Ω]	Resistance
I	[A]	Current
σ_{max}	[MPa]	Maximum stress
σ_{min}	[MPa]	Minimum stress
σa	[MPa]	Alternating stress
R	[MPa]	Range of stress
σm	[MPa]	Mean stress
ΔK	-	Stress-intensity factor
A	[mm]	Crack length
m	-	Constant (materials parameter)
n	-	Constant (materials parameter)
C	-	Termed material constant

1. Introduction

Welding is efficient and economy process. Thus, widely practiced the way of joining of metal and it is unquestionably the oldest method for the joining materials. Welding is, in fact, an ancient art. [1]

When all are talking about welding there is one question comes to mind “what is welding”? “Welding is a process in which materials are brought together and cause to permanent join through the formation of primary atomic bonds”. There is possible to use only the heat, combined action of heat and pressure or only pressure to create joint. This process is possible to use with or without of filler metal.

The welding divided such essential ways: [1]

1. In the welding, process continuity implies the absence of any physical disruptions of atomic scale, that is, no gaps.
2. Welding is a versatile process; it applies not just to metal. It can and often does apply equally well to plastic (e.g. thermoplastic) materials.
3. Welding is not always combined action. It depends upon which physical principle going to apply to the welding process. There is a widely held perception that welding is performed by melting of materials but there is also possible to weld materials in the solid state. Welding is a highly flexible joining process where are created homogenous or heterogeneous welds to obtain different properties for many different purposes. The essential point for certain welding technologie is that an intermediated or filler meterial. Filler material requries may or may not be the same composition as like base materials.
4. Welding is a secondary manufacturing process used to produce an assembly or structure from parts or structural elements.

The ideal welding is when atoms are bonded at the fundamental bases. To produce an ideal weld is to bring atoms together to their equilibrium spacing in large numbers to produce joints without any gap and with the same level of strength as the base metal. [2]

There are currently 94 different welding technologies based on over 30 different physical principles. On Fig. 1 there is a distribution of basic welding methods. This master thesis is focused on solid state welding specifically on diffusion welding.

This process is able to help make high precision components with complex shapes with the flexible working condition. Diffusion welding is used for demanding industries for reason that is high quality welds, deep and narrow profiles, limited heat affected zone and low thermal distrotion. The biggest positive is that is possible to join dissimilar metals (difference in physical, thermal and chemical properties) with the very small heat affected zone.

According to this process is not requried any liquid phase and frequntly no filler metal. Welding processing time does not depend on specimen size. [1,2,3]

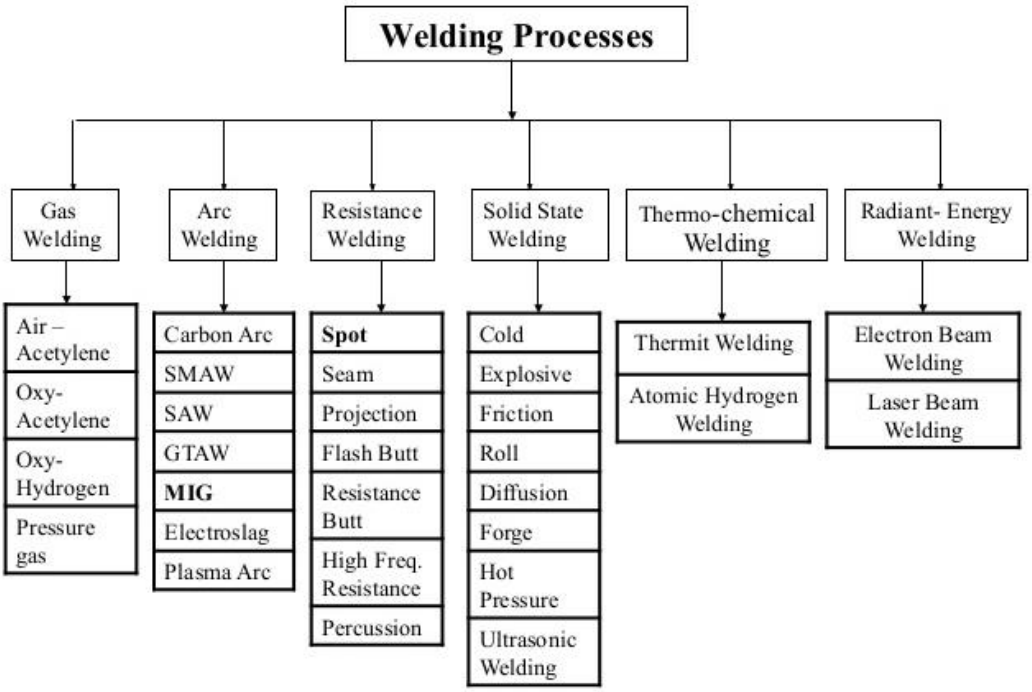

Fig .1. Classification of welding process [4]

2. Theoretical part

2.1. The principle of diffusion welding

Diffusion welding is a process, which works on the fundamental principle of diffusion. Diffusion means movement of molecules or atoms from high concentration region to low concentration region as is shown in Fig. 2. [6] This is a basic principle of diffusion welding. In this process, two or more the welding plates are placed one over other for a long period of time under high pressure and temperature. Thanks to pressure force start diffusion between interface surfaces. By the help of high temperature, the diffusion process can accelerate, but temperature does not melt the welding plates. The temperature range is about 55 - 90% of melting temperature. This whole process takes place in a vacuum or in an inert environment which shields the welding plates from oxidation. [2]

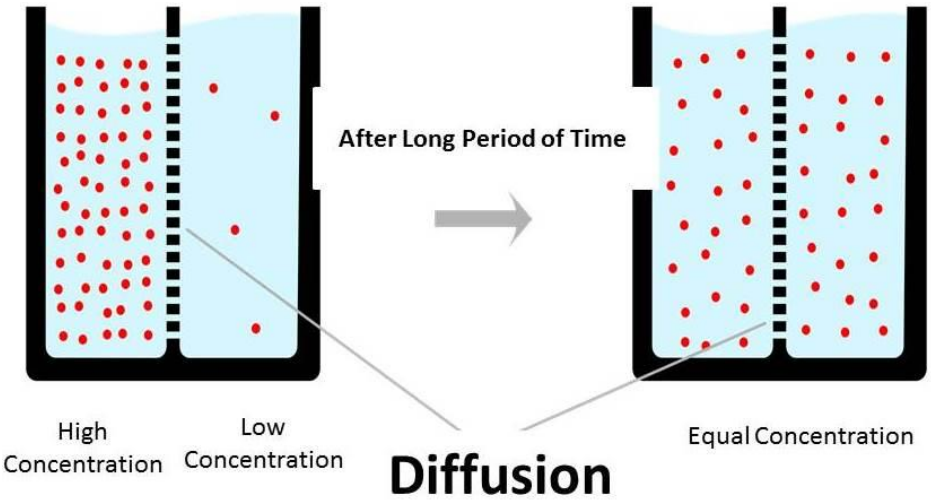

Fig. 2. Molecules or atom movement in diffusion process [4]

During the process, at first, the welding plate surfaces have to be prepared for welding. The interface surfaces made equally flat which is a basic necessity of diffusion process. The interface surfaces should be machined, grinding, cleaned and polished well which expel all chemical contaminants from the surface. Any contaminant particle can be minimizing diffusion between welding plates. [2,4]

After that, the plates are clamped and put one over another. This organised placed into a vacuum chamber or in an inert gas environment. It protects the welding surface from oxidation. For begin diffusion on assembly, the section must be applied pressure and temperature. The temperature applied by the furnace heating or electric resistance heating.

[5] The pressure is transferred by a hydraulic press, dead weight or by the differential gas pressure. These conditions are maintained for a long duration of time for appropriate diffusion. [1]

Diffusion welding process is schematically shown in Fig. 3. Initial surface contact is very small due to roughness (Fig. 3 a). At the beginning stage of the process is used press force. It will cause local deformation at the interface surface due to creep and yield. The touch area is increasing and the diffusion takes place which forms an interface boundary (Fig. 3 b). Due to temperature, pressure and time, diffusion is running and gaps between surfaces are slowly disappearing (Fig. 3 c). After a defined period of time, the plates are properly diffused into one another which make a strong solid joint. The interface boundaries vanish from welding area which forms a clean joint (Fig. 3 d). This joint should be having identical properties or strength as the base material. [1]

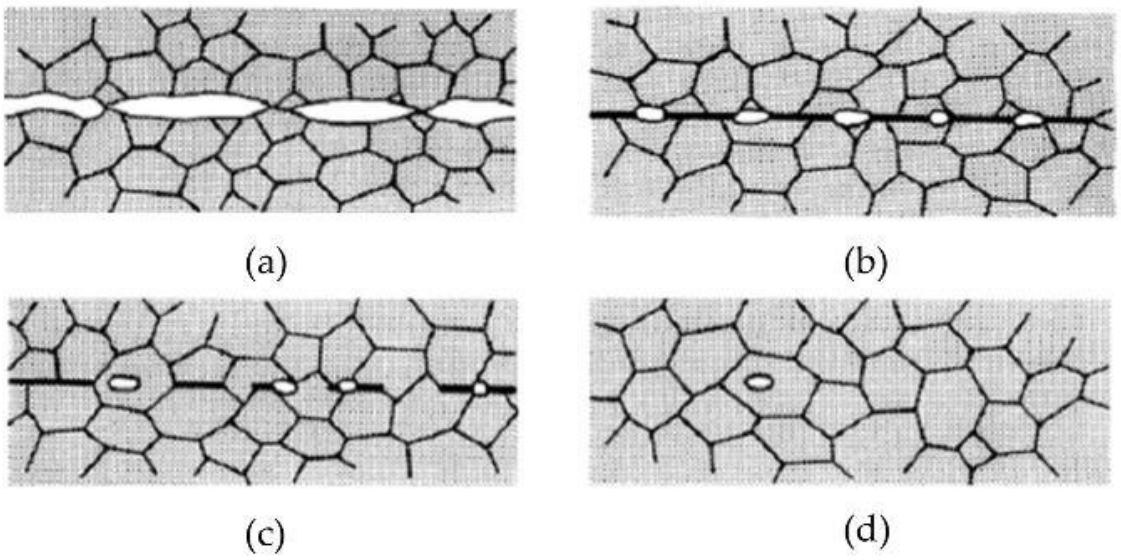

Fig.3. Basic phases creation of diffusion joint [6]

2.2. Diffusion Theory

Diffusion theory regards internal composition of material including crystal lattice interferences. In case of non-existing motive power on atoms, their jumps are random. The surface migration of metallic atoms shows low activation energy as well. The knowledge of concentration is summarized by the two Fick's laws. According to the first Fick's law eq^{n}. (1), the diffusion flow J_A of element A atoms throughout given time, in the direction of axis x and throughout the surface unit depends on the gradient of element concentration $\frac{\partial C_A}{\partial x}$ and is

proportional to the diffusion coefficient D. Simply started, the first Fick's law expresses rate and amount of atoms which diffuse through the material. [4,6]

$$J_A = -D\,(dc/dx) \qquad (mol.m^{-2}.s^{-1}) \qquad (1)$$

Where:

C_A	-	is concentration of element A	($mol.m^{-3}$)
X	-	is direction of diffusion concentration change	(m)
t	-	is amount concentration change time	(s)
D	-	is diffusion coefficient	($m^2.\ s^{-1}$)

The quantity of diffusing material *q* is a function of the concentration gradient of the element engaged, dc/dx, in a direction normal to the interface Fig. 4 and is proportional to the diffusion coefficient, D. [4]

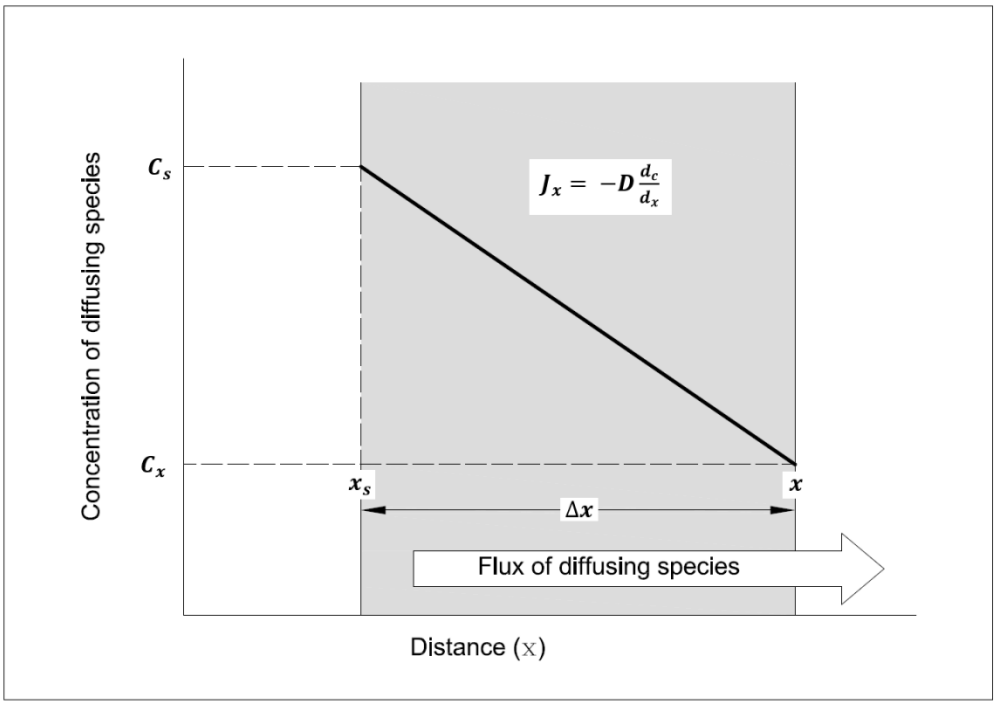

Fig. 4. Concentration of diffusion vs distance

Fick's second law in eq^n. (2) estimate how diffusion causes the concentration to change with time. It is a partial differential equation which in one dimension. [1] There is termed so-called non-stationary diffusion.

$$\frac{\partial C_A}{\partial t} = \frac{\partial}{\partial x}\left(D\frac{\partial C_A}{\partial x}\right) \qquad (mol.m^{-3}.\ s^{-1}) \qquad (2)$$

Where:

C_A	-	is concentration of element A	($mol.m^{-3}$)
X	-	is direction of diffusion concentration change	(m)
t	-	is amount concentration change time	(s)
D	-	is diffusion coefficient	($m^2. s^{-1}$)

Diffusion of atoms should be divided into two groups such as self-diffusion and hetero-diffusion process. When diffusion occurs without any presence of driving force, it is called self-diffusion. Hetero-diffusion related to diffusion occurs with help of reactants.

The diffusion (both self- and hetero-diffusion) of atoms can take place only if the diffusing atoms have an adequate store of energy for their migration through the lattice. At any temperature, the mean energy of oscillation of atoms in the lattice is a proper quantity. This energy, however, varies from atom to atom in accord with laws of probability. [4]

As is shown in Fig. 5 the movement of atoms can be accomplished in four ways. First possibility (a) is changing the two adjacent atoms. The second possibility (b) is a transfer of vacancies (there is a mutual exchange of vacancies with adjacent atoms). Next possibility (c) is atom penetration between individual nodes and the last possibility (d) is exchange 4 atoms in a circle. [4,5]

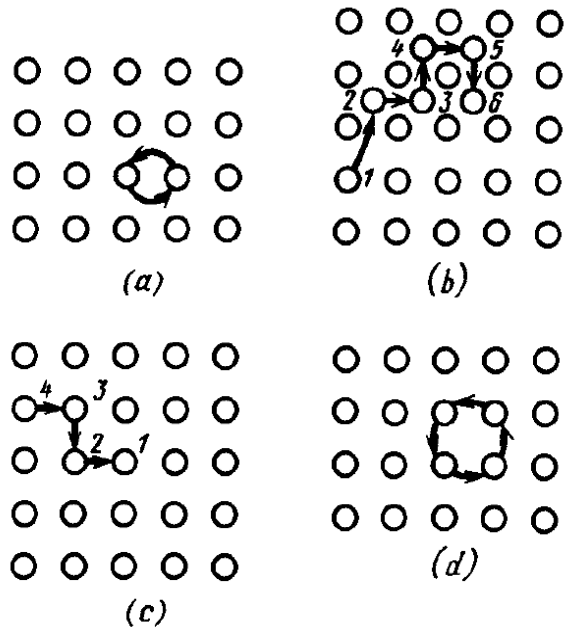

Fig. 5. Possibilities of atom movement in lattice a) Changing two adjacent atoms; b) Transfer of vacancies; c) Atom penetration; d) Exchange 4 atoms on a circle [6]

2.2.1. Hypotheses of diffusion

Some hypotheses have been progressed to explain how a bond is formed in the solid state. There are currently 6 hypotheses that can be used to explain the diffusion process. They are film hypothesis, recrystallization hypothesis, energy hypothesis, dislocation hypothesis, electron hypothesis and diffusion hypothesis.

By **the film hypothesis**, all metals and alloys possess the same property to seize, when clean surfaces are brought together within the range of interatomic forces. The observed differences in weldability among various metals are clarified by the presence of surface films**.** The oxide films which are bad for joining can be hard, brittle, viscous, or plastic. When the metals being joined are subjected to cold plastic deformation, the hard and brittle films are broken up to reveal clean metal layers which, on being closed together within the range of inter-atomic forces, form a strong bond. In all the cases oxide film was played the minor role in bonding. [4]

The recrystallization hypothesis is put important on recrystallization as the principal factor in bond formation. By this hypothesis, deformation and the following strain hardening, coupled with exposure to relatively high temperatures at the interface, because the atoms in the lattices of the materials are being joined to flow to other sites so that there appear, at their boundaries, grains common to both pieces with the result that a bond is formed. Recrystallization formed the new grain in welding area, from the strong bond. [4]

The energy hypothesis*,* for a diffusion bond to form the atoms of the metals being joined, should be raised to what may be called the energy threshold of adhesion. At this threshold, the formation of atomic bonds is not an important factor, metallic bonds come into being between the atoms at the surfaces, and the interface between the two pieces disappears. The combination of plastic deformation important for the onset force applied to the metal decreases with increase in the energy of an atom of the metal. The energy hypothesis fails to derive which properties of the metals being joined are responsible for the degree of force to make bonding. [5,6]

By the dislocation hypothesis due to J. Friedel, E. I. Astrov, and some others says that “the joint plastic deformation causes dislocations to move to the surface”. One body of opinion is that the introduced of dislocations at the contact surface minimise resistance to plastic deformation and aids in joining the metals. Bond formation is a result of the plastic flow of the metal within the contact zone**/**welding zone. [2,5]

The electron hypothesis has been advanced by G. V. Samsonov et al. In their opinion, “the surface pressure results in the formation of stable electron configurations involving the

atoms of the metals in contact". If the electron configuration of two metals having a high weight in statistically the bond strength or adhesion must be a lower strength. The electrical configuration of metal and element factor gives on sight into their weld ability, wettability, diffusion processes, etc. [1,5]

By **the diffusion hypothesis**, the formation of a good bond between the surfaces in contact based on the inter-diffusion of atoms into the dimension of the specimens. The surface atoms of a metal have free, unfilled bonds (vacancies) which capture any atoms moving within the range of inter-atomic forces. A high concentration alloy joint with low concentration alloy after the diffusion with the help of inter atomic force both alloy having equal concentration. [5]

2.3. The activation energy of diffusion

Activation energy is important for atom movements inside the lattice structure. During interstitial diffusion, there is a chance that the neighboring sites are vacant. However, in substitutional diffusion, it is bit complicated, since vacancy should be present in the next neighbor position and then only there will be a possibility of the jump. Here is talked about activation energy required for interstitial self diffusion. [3]

Atom from the ground state (marked as G_g) jumps to another ground state but go through an activated state (marked as G_a) in Fig. 6. where it has to move its neighboring atoms elastically and then it should move to one more place as a plastically and energy level should be again G_g. [3]

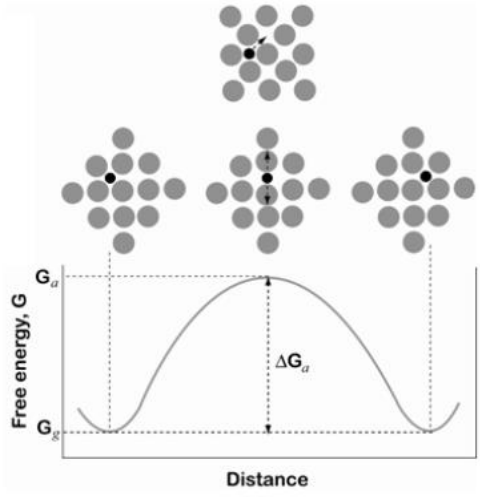

Fig. 6. Free energy vs atom reversibly move distance [3]

Activation energy can be evaluated from the diffusion coefficients, calculated at different temperatures according to the eq^n. (3), respectively according to the eq^n. (4).

$$D = Do \exp\left(-\frac{Q}{RT}\right) \quad (3)$$

$$\ln D = \ln Do - \frac{Q}{RT} \quad (4)$$

Where:

D	-	Diffusion coefficient	($m^2.s^{-1}$)
D_0	-	Pre expontial factor	($m^2.s^{-1}$)
R	-	Gas constant 8,3144598	(8.314 J/mol k)
Q	-	Activation energy for diffusion	(J)
T	-	Temperature	(°C)

The result is plotted in graph D vs. 1/T as is shown in Fig. 7. From graph (slope) is possible to determine the activation energy which is necessary for diffusion, Q. [3]

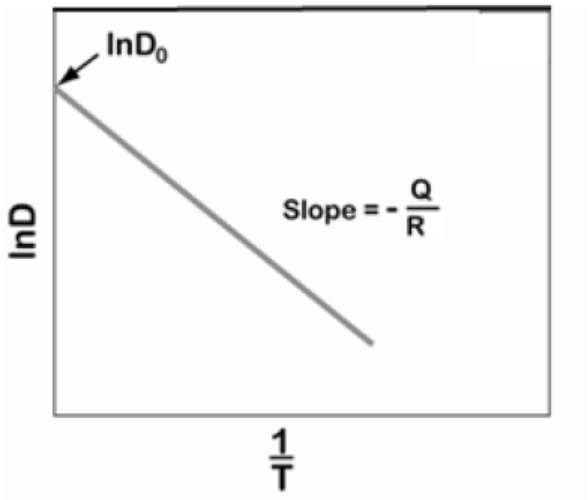

Fig.7. Diffusion coefficient vs temperature [8]

2.4. Bonding mechanism

Generally, different types of bonding mechanisms are used for the diffusion process. These are according to the sequence of occurrence: (1) plastic yielding resulting in deformation of original surface asperities, (2) next surface diffusion from surface source to a neck. Then follows volume diffusion from surface source to a neck (3) and disappear from a surface source to condensation at the neck (4). Next steps are grain boundary diffusion from an inter -facial source to a neck and volume diffusion from interfacial source to a neck (6). The last one is a creep at a modest temperature (7). [9,10]

All these mechanisms are separated into three main parts:

Stage 1 - Plastic deformation. The contact area between two surfaces is very small, having asperity, initially small when applies pressure and temperature quickly grow the contact surface area, which means local stress below the yield strength of the material. Some factors are very important for the first step of bonding such as surface roughness, yield strength, hardening after machining, temperature, and pressure. Stage 1 called as high pressure diffusion welding phase is schematically explained in Fig. 8. [11,14]

Fig.8. Surface structure before and after welding [14]

Stage 2: During the second stage, creep and diffusion role more than deformation and many of the voids shrink and some of the voids are disappeared as grain boundary diffusion of atoms continues. [9,14]

Stage 3: In the third stage, the remaining voids are removed by volume diffusion of atoms to the void surface.

For good bonding required a proper combination of flatness and smoothness of the surface. A certain minimum degree of flatness and smoothness is required to guarantee uniform contact. Recrystallization plays the important role in surface diffusion, which increases the speed of the diffusion. [9,14]

The various routes for diffusion are contained in Fig. 9 and these mechanisms are divided into two main stages. Stages of deformation and diffusion and power law creep. The atoms start to migration provides the basic class of mechanisms by applies pressure and temperature simultaneously. The mechanisms are in Fig.9: (1) plastic yielding resulting in deformation of original surface asperities; (2) surface diffusion from surface source to a neck; (3) volume diffusion from a surface to a neck; (4) evaporation from a surface source condensation at a neck; (5) grain boundary diffusion from an interfacial source to a neck; (6) volume diffusion from an interfacial source to a neck; and (7) diffusional creep under the action of capillary force. [9,10,14] In other words, no fundamental distinction needs be made between stress induced matter transport (coble creep) that giving from the presence of a curved interface. [13]

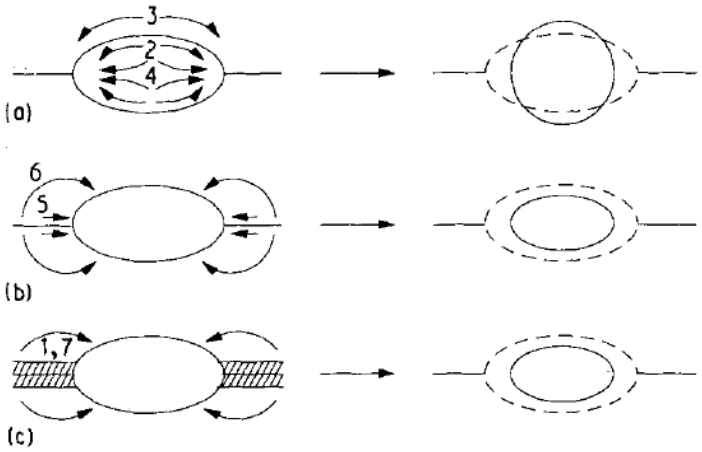

Fig 9 various mechanisms of materials transfer [13]

The Fig (9) explains that diffusion of the atom through the surface source, Fig (9.b) material transfer through interface source and Fig (9.c) the bulk deformation of mechanism [13].

2.5. Fundamental process parameter

Diffusion welding depends upon the certain parameters and parameters assembled into six categories: (1) surface preparation, (2) temperature, (3) time, (4) pressure, (5) special metallurgical effect and (6) using of the interlayer. Highly important and main process parameters are temperature, time and pressure. [8]

The influence of **temperature** on the diffusion process should be characterized in the following points:

1) Temperature is possible promptly changed and easy to measure and control.
2) Temperature has an impact on plasticity, diffusivity, oxide solubility, etc.
3) Temperature has an influence on allotropic transformation, recrystallization and other actions in materials.
4) Increasing temperature increases a diffusion rate and it allows decreasing of welding cycles. It has an influence on economic of the operation.
5) The temperature should be higher than 0.5 melting temperature T_m. It correctness in between 0.6 to 0.8 melting point.

Also, the influence of **pressure** on the diffusion process should be characterized in the following points:

1) Pressure affects several of the diffusion welding mechanisms. The initial deformation phase of bond directly affects the intensity of pressure applied.

2) Higher pressure means greater interface deformation and lower localized recrystallization temperature.
3) Pressure should be kept up constant during all welding process (holding time).
4) The level of pressure heavily depends on the used temperature and on the mechanical properties of the welded materials.
5) Press is for a given temperature level evaluating with help of cascade (RAMP) test.

The influence of **time** on the diffusion process should be characterized in the following points:

1) Time depends on temperature and pressure because diffusional reaction linearly or parabolic related to time. An increase in temperature compresses the amount of time required to complete a diffusion process. [6]
2) In the system with thermal and mechanical inertia, diffusion time is longer due to the unreasonably of a suddenly changing variable. If there is no inertia, the problem may be a welding time reduction.
3) For conservative point of view, it should be reduced welding time factor so than it can increase the production rate.

2.6. Surface roughness and surface preparation

The review of surface roughness decides the degree of beginning surface contact and the estimate of voids. This, in turn, influences the bonding rate. Surfaces may be arranged by machining, grinding and polishing. In general, a surface roughness better than around 0.4 μm is necessary to ensure good initial contact. The removal of surface contaminants and thick oxides prior to bonding is also crucial. [6,8,12]

The initial surface finish is simply obtained by machining, grinding or polishing. An accurately prepared surface is flat. Flatness and smoothness are essential in order to assure that the interface can achieve the necessary compliance without an excessive level of deformation at welding zone. [8]

Machine finishes, grinding or abrasive polishing is usually adequate as long as an appropriate precaution is exercised to minimise warpage and distortion. [5]

The secondary effect of the initial machining or abrading, not always recognized, is the deformation established into the surface during machining. [5,6]

In diffusion bonding, the oxide film is also a big issue to obtain high quality joining. There have been reported several solutions to this issue. They are: inserted inter layer, surface treatment, carried out diffusion bonding in a vacuum or an inert gas such as Ar. The surface treatment, especially grit blast, is the most famous method in this solution. Before diffusion

bonding, grit blast treatment is performed to expel the surface oxides and supply adequate rugged surface. [5]

In Fig. 10 there is shown the effect of roughness on weld strength by diffusion welding of steels 12 060 and 19 463. In this case, was used at temperature 950 °C, press 20 MPa and welding time 300 sec.. From graph his clear that the high strength was achieved in the range of roughness R_a from 1,6 to 3,2 μm.

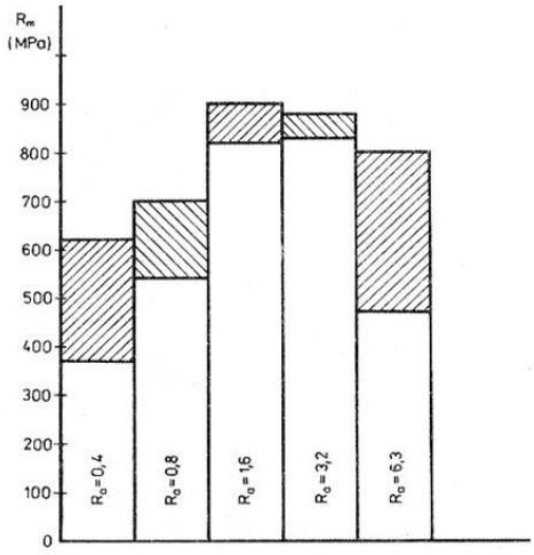

Fig. 10. Effect of roughness weld strength for welding steels 12 060 and 19 463 [5]

2.7. Method of heating material during diffusion welding

The various processes of heating the work pieces during diffusion welding can be divided into two groups. In the first group, the heat is transferred to the work piece through conduction or radiation thanks to an external heat source. The other one used the heat which is produced in the work pieces themselves by the conversion of electricity into thermal energy. It is especially at the contact point of the materials where the greatest transient resistance. [5]

2.7.1. Radiation heating

A heat source may be situated inside or outside the work or vacuum chamber. The highest acceptable temperature for radiation heating depends on the thermal stability of the chamber material. In diagram form, several postioning using radiation heating are shown in Fig. 11. The workpiece, 1, is set up on the amount, 2, inside the vacuum chamber, 3, and is heated by radiation from a heater,4, placed outside (Fig. 11a) or inside (Fig. 11b). The heating rate

can be controlled by varying the voltage applied to the heater. In practice, the heaters, 4, are usually placed inside the chamber. [5]

The above process some demerits also there, where heater material might vapour and stick on the surface of the workpiece. This can be eliminated by (Fig. 11.c), where S_0 that the work piece could not melt and be welded to the heater if they were in direct contact, the latter is given a thin coat of aluminium oxide, 4, which separates them.

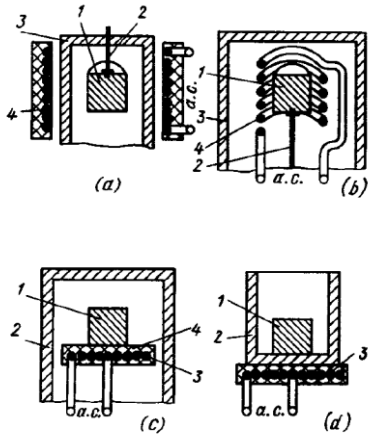

Fig. 11 Heating parts by radiation and conduction [5]

In the arrangement shown in (Fig. 11 d), the workpiece, 1, is placed inside the chamber, 2, and heat is supplied by an electric furnace, 3, situated outside. Now heats usually supplied by thermal conduction, moreover, radiation from the surface of the hot chamber also plays a crucial part.

2.7.2. Resistance heating

During resistance heating, the essential heat is supplied by the passage of an electric current through the work pieces themselves. The pieces are in direct contact with the current-source. The rate of heating determines the resistance of the specimen R_s, and the rms value of the current I_{rms}, passing through the specimen. The amount of heat, Q (J), generated by the movement of current can be found by Joule's law - eq^n. (5):

$$Q = I_{rms}^2 . R_s . t \quad (5)$$

Where

Q	-	Amount of heat	(J)
t	-	time	(sec)
I_{rms}	-	Value of current	(A)
R_s	-	Rate of heating	(W)

In resistance heating, the higher temperature of the work depends upon only its melting point. An important requirement for resistance heating is an arrangement of a reliable physical contact between the work and the electrodes conveying the current. [3-6]

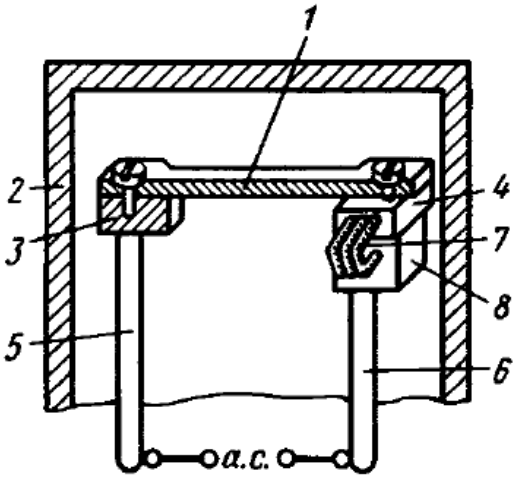

Fig. 12. Resistance heating [5]

An arrangement for resistance heating is shown in Fig. 12. The workpiece, 1, placed inside a vacuum chamber, 2, is clamped in jaws, 3 and 4. Terminal 3 is made fast to an electrode, 5, whereas terminal 4 is connected to a second electrode, 5, by a copper pig-tail, 7, and a copper block, 8. Provision of a flexible pig-tail is important as it avoids straining the work due to the volumetric changes occurring in the course of heating and cooling. [2,5]

Apart from copper, the electrodes can be also made of graphite and tungsten, in which case the materials to be heated may broadly vary in thermal conductivity and resistance. A further advantage is that heat can be provided to hard-to-reach spots. The best type of electrode for a given material can be established by experiment, using various combinations and various geometry. With graphite or carbon electrodes which are generally soft, a minimum pressing load should be used than in the case of electrodes made of high-temperature alloys and steels. [5]

2.7.3. Induction heating

In this case, the workpiece to be heated is put in the high-frequency electromagnetic field set up around an inductor by a source of high frequency current. A refinement of induction heating is that electric energy is transferred from the inductor to work over a distance of a few centimetres, without any definite contact between them. Heat is generated within the work by the circulating eddy currents induced by the applied magnetic field. [5] The current thus induced is possible express by eq^{n}. (6):

$$I = E/R \quad (6)$$

Where,

R - total apparent resistance (Ω)

E - Electro motive force (V)

I - Current (A)

2.8. Thermal-stress simulator Gleeble 3500

The temperature-stress simulator Gleeble 3500 is a product of the American company Dynamic System Inc. and it is used to test material response during various mechanical and metallurgical conditions. Physical simulations of technological processes are being used nowadays more often.These simulations serve to simulate the thermal-mechanical processes which correspond to real conditions, but they are performed in laboratories. Simulator Gleeble 3500 was purchased by the Technical University of Liberec in 2013 and is used especially for the testing of forming and welding processes under different temperature-stresses conditions. The Gleeble device will be used to solve the practical part of this thesis. Fig. 13 shows the basic Gleeble 3500 simulator assembly.

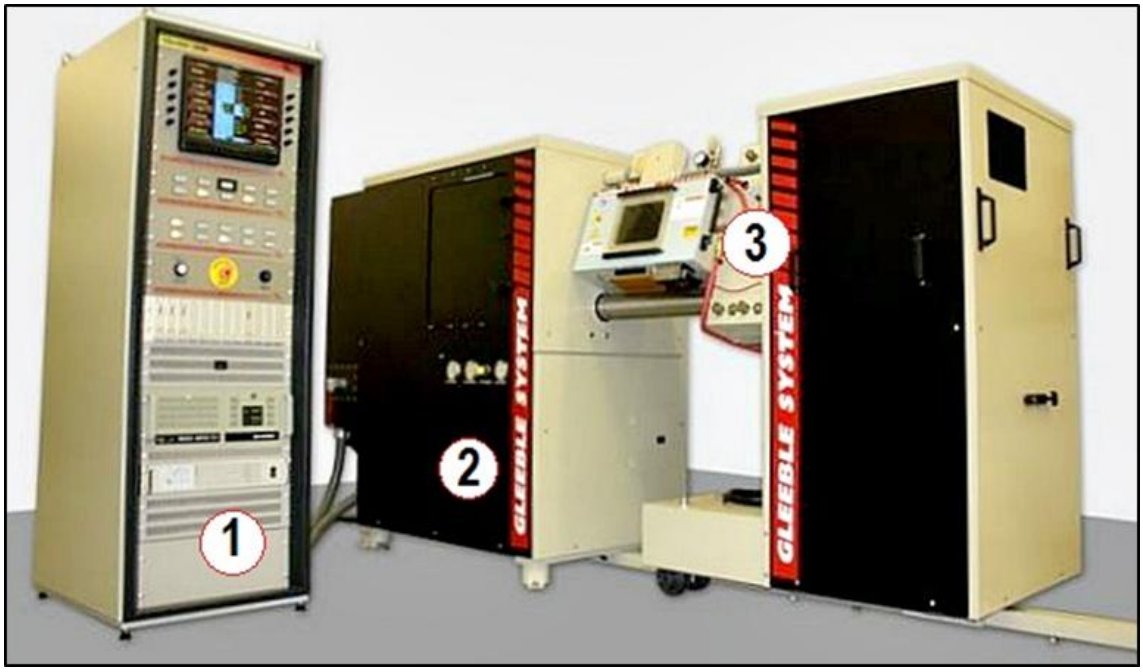

Fig. 13. Basic Gleeble 3500 simulator assembly. 1- Console; 2- Load unit; 3 – Pocket Jaw

2.8.1. Basic information

The Gleeble 3500 is the most commonly used temperature-stress simulator. This dynamic system can be used to identify almost all of the happenings run in metals at high temperatures. The device library was created over 50 years and therefore contains a huge amount of information about the machine. Device Gleeble can simulate almost any thermal-mechanical load that occurs both during processing and during subsequent operation.
The Gleeble system is capable of testing samples with a maximum diameter of 20 mm or with a cross-section of up to 400 mm^2. The device can create maximum tensile or compressive force of 100 KN and the maximum heating rate is more than 10,000 °C S^{-1}. Maximal the cross beam speed is up to 1 $m.s^{-1}$ and stroke size up to 100 mm. Maximum numbers and the frequency of the compression moves depends on the magnitude of the load force and on the simulated event.

2.8.2. Tempearture system

The direct resistance heating system in Gleeble can heat specimens at rates of up to 10,000 °C. S^{-1} or can hold steady state equilibrium temperatures. High thermal conductivity grips hold the specimen, making Gleeble capable of high cooling rates. The clamping jaws are used both to heat and to cool with the test sample. When combined with other cooling devices, it is possible to cool the surface of the sample up to 6,000 °C. S^{-1}. This way of heating can do keep the required heating and storage temperatures accurate to ± 1 °C. The Gleeble heating rate is many times higher than the pressure load.
There are two ways of controlling and monitoring the temperature of the specimen during heating and cooling. One is by thermocouples and the other one by an optical pyrometer. There are four thermal channels available in a standard Gleeble system, with either four thermocouples channels or one pyrometer channel and three thermocouples channels. Different thermocouples can be used depending on the temperature range of the test. It is possible to choose from types B, E, K, R and S. One of the most widely used types of thermocouples is type K because it has a wide operating range from -180 °C to +1250 °C. For very high temperature is using an R-type thermocouple. It is operating range up to 1450 °C. Thermocouples are welded to the test sample in most cases with help of capacitor welder. Before welding are important samples to clean and degrease properly. Fig. 14**.** shows connection points for measurement of temperature.

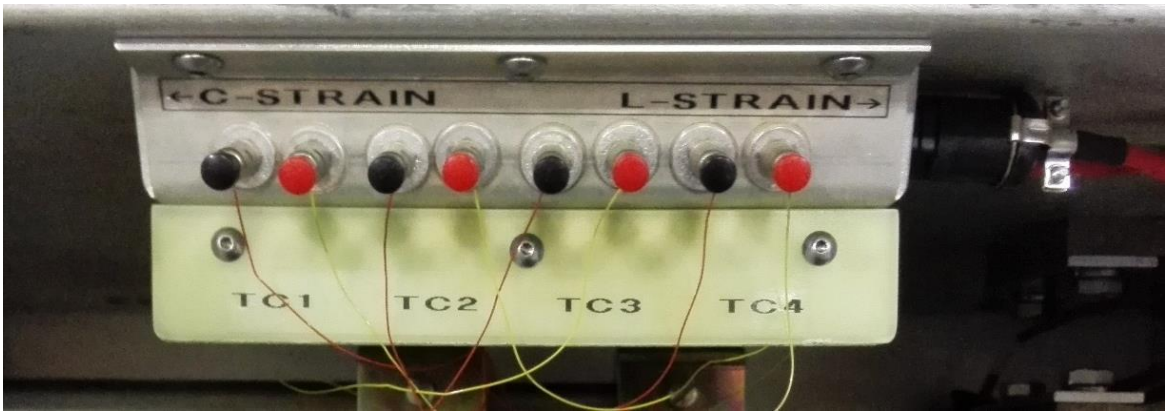

Fig.14. Channels of four thermocouples

2.8.3. Temperature gradients in the sample during simulation

The Gleeble device allows to control the temperature gradient patterns in the samples during the simulation. In cases like welding processes to which, of course also includes diffusion welding, working with a steep temperature gradient to degrade material in the vicinity of the joint as small as possible. In other cases, it is required for uniform temperature effects and flat temperature gradients.

The temperature gradients of the test sample are affected by the type of test material, its electrical and temperature resistance, the free length between jaws, cross section and the ratio of the surface to the total volume of the component. Free sample length represents the distance between the edges of the high temperature clamping jaws. It also applies that the longer it is contact between the sample and clamping jaws, the steeper the temperature gradient inside of the test sample. Extending the distance between the specimen and the jaws increases differences between the maximum and minimum temperatures achieved in the sample.

In practice, jaws with partial or full contact are used and theoretically it is possible to use any material to produce them. The most used materials for production these jaws are copper (containing Cu 99%) or austenitic high - alloy X5CrNi18-8 steel. There are considerable differences in the thermal conductivity of both materials; therefore, they have very different temperature gradients. Fig. 15. shows a temperature gradient on a sample of S355J2 steel in use copper jaws with full contact and free length 30 mm. It is created for a temperature range from 200 to 1200°C. Fig. 16 shows a temperature gradient on a sample of S355J2 steel in use X5CrNi18-8 steel jaws with full contact.

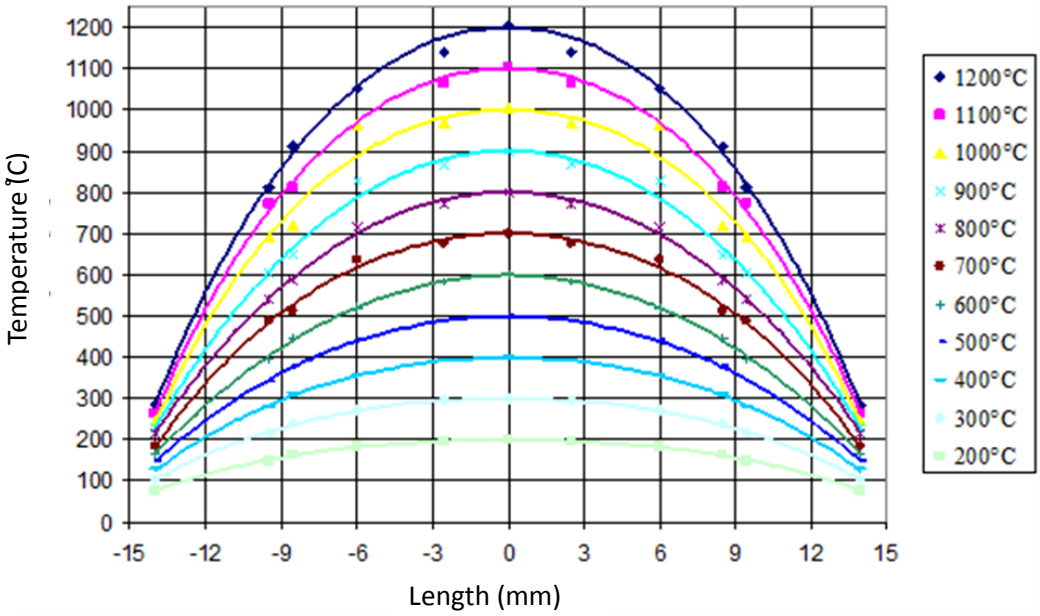

Fig. 15. Graphical representation of the temperature gradient over the 30 mm free length of the S355J2 steel sample when using full-length copper jaws

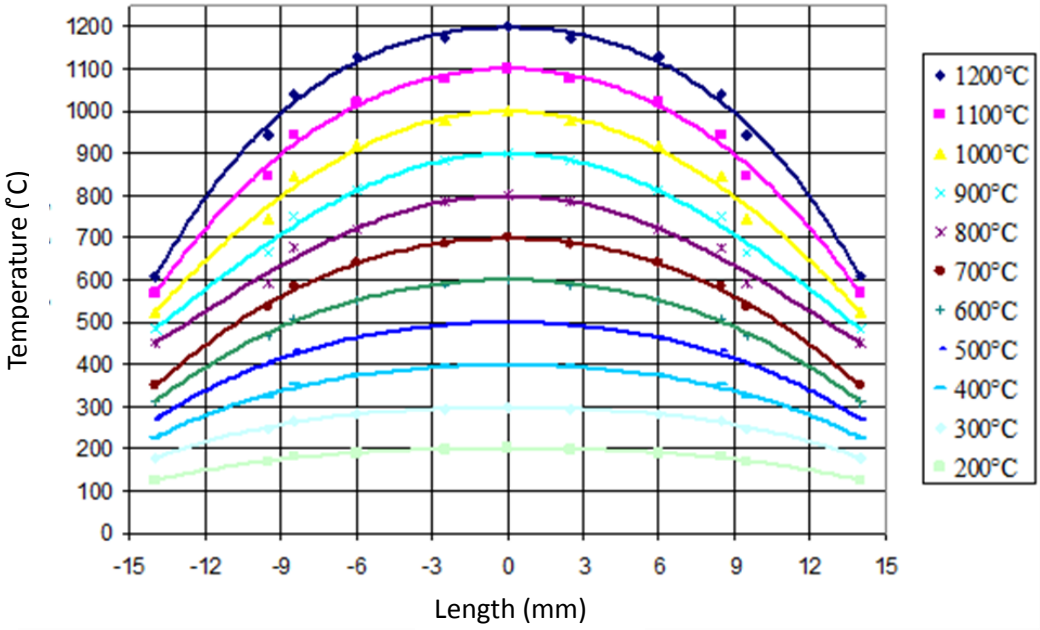

Fig. 16. Graphical representation of the temperature gradient over the 30 mm free length of the S355J2 steel sample when using full-length X5CrNi18-8 steel jaws

Fig. 17 shows the different types of high temperature jaws used in the Gleeble instrument, both in terms of the material used and the shape aspect the size of contact between the jaws and the samples.

Fig. 17 Jaws with partial and complete contact made of copper and X5CrNi18-8 steel intended for different shapes of test samples

2.9. Advantages and Disadvantages of Diffusion welding

As every technological process, also diffusion welding has its own advantages and disadvantages both in terms of process and economic aspects. The main advantages of this method are:

1) Diffusion joints have almost the same mechanical and physical properties as the parent material.
2) This process produces a clean joint that is free from interface discontinuity and porosity.
3) Both similar and dissimilar material can be joints by diffusion welding process. It means, it is possible to create homogenous and also heterogeneous welds.
4) It provides good dimension tolerance. So it is used to make precision (final) components.
5) The joining is in the solid state, in welding area, there is not casting structure.
6) The process causes only very small deformations.
7) It is simple in working.
8) There is not used filler material, flux, etc. which are used in the arc welding process.
9) It is possible to weld complex shapes together.

The main disadvantages of this method are:

1) High initial or setup cost.
2) It is a time consuming process. It takes more time compared to other welding processes.
3) Surface preparations of welding plates are more expensive and difficult.
4) Size of the weld is limited according to equipment available.
5) This process is not suitable for mass production.
6) Highly depend on welding parameters like surface finish, welding material, temperature, pressure etc.

3. Fatigue and ways to test it

Fatigue properties of any engineering materials are very important for mechanical engineering practice. Fatigue is the progressive, localized, permanent structural change that occurs in materials subjected to fluctuating stresses and strains that may result in cracks or fracture after an adequate number of fluctuations. Fatigue fractures are caused by the simultaneous action of cyclic stress, tensile stress, and plastic strain. If any one of these three stress is not present, fatigue cracking will not begin and propagate. The cyclic stress starts the crack; the tensile stress produces crack growth (propagation). Although compressive stress will not cause fatigue, compression cyclic load may do so. [16]

The process of fatigue consists of three stages:

• Starting fatigue damage leading to crack nucleation and crack initiation. [16,17]

• The progressive cyclic growth of a crack (crack propagation) until the remaining un-cracked cross section of a part becomes too weak to sustain the loads imposed. [16]

• Final, quick fracture of the remaining cross section.

Fatigue cracking regularly results from cyclic stresses that are well below the static yield strength of the material. (In low-cycle fatigue, however, or if the material has an appreciable work-hardening rate, the stresses also may be above the static yield strength). [16,18]
Fatigue cracks initiate and propagate in regions where the strain is most serious (where the largest notch effect). Most engineering materials have some defects and thus area of stress concentration that intensify strain. Most fatigue cracks initiate and grow from structural defects. Under the action of cyclic loading, and a plastic zone (or region of deformation) develops at the defect tip. [18] This zone of high deformation becomes an initiation site for a fatigue crack. The crack propagates under the applied stress through the material until total

fracture results. On the microscopic scale, the most important feature of the fatigue process is nucleation of one or more cracks under the impact of reversed stresses that exceed the flow stress, tracked by the development of cracks at persistent slip bands or at grain boundaries. [17-19]

Cyclical stresses (Fig. 18) can take place entirely in the area of tensile stresses, at the boundary between tension and pressure, or entirely in the area of pressure. The magnitude of the oscillation amplitude and the mean level of the stress are determined by the area of the load movement.

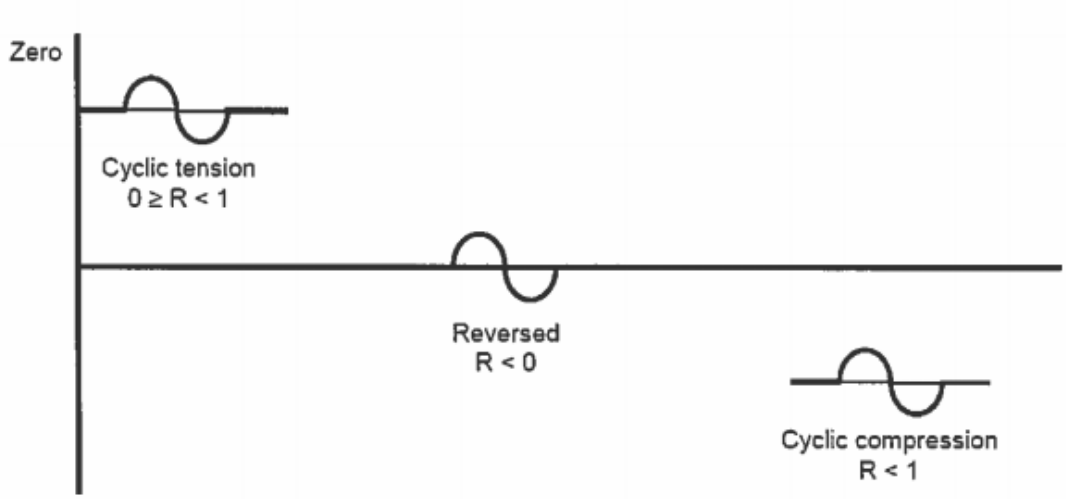

Fig. 18. Cyclic stress at different zones

There are generally recognized the three main types of load cycles. They are **purely cyclic tension, purely cyclic compression** and **reversed** (symmetrical and asymmetrical) cycles.

Purely cyclic tension - the stress/load oscillation may be sinusoidal, but the mean stress/load must be such that the stress state during the entire cycle is tensile. Needless to say, for a given stress amplitude this type of loading is more severe (σ_{min} is a zero or in a tension area).

Purely cyclic compression - the stress/load oscillation may be sinusoidal, but the mean stress/load must be such that the stress state during the entire cycle is compression (σ_{max} is a zero or it is in a compression area).

Reversed cycle – it is the simplest loading one can conceive is a sinusoidal wave pattern loading, where the stress/load oscillates about a mean zero load/stress. In the case of an asymmetric cycle, the mean stress value is not zero, but the cycle must extend to both areas (stress, compression).

Random stress cycles – These cycles generated in real-time and real-load cycles. They are generated under different boundary conditions. As a rule, they do not have an exact sine wave load. An example of random stress cycle is shown at Fig. 19

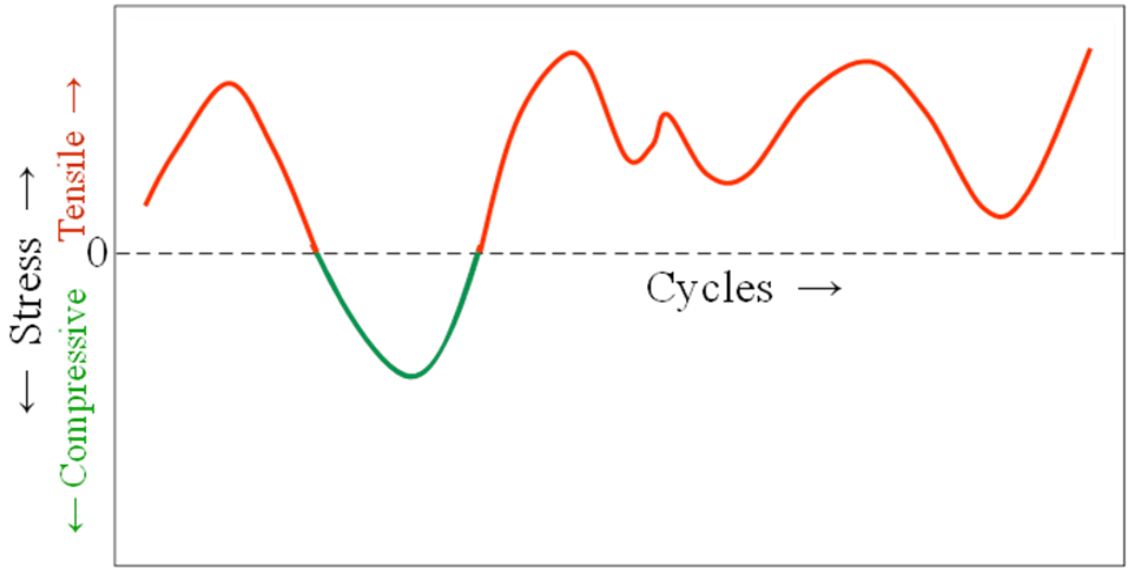

Fig. 19 . Random stress cycle [20]

The stress cycles that are used in fatigue studies are characterized by many parameters, such as mean stress, alternating stress, stress ratio and amplitude ratio. The range of all varies is between σ_{max} (Maximum stress) and σ_{min} (Minimum stress). [17,18]
The individual variables are defined as follows:

The range of stresses - $\sigma_r = \sigma_{max} - \sigma_{min}$

Alternating stress - $\sigma_a = \sigma_r/2$

Mean stress - $\sigma_m = (\sigma_{max} + \sigma_{min})/2$

Stress ratio - $R = \sigma_{min}/\sigma_{max}$

Amplitude ratio - $A = \frac{\sigma_a}{\sigma_m}$

3.1. Factors for fatigue fracture

Three basic factors are necessary to cause fatigue failure. These are:

- Sufficient high value stress which gives maximum tensile stress.
- A large enough difference or fluctuation in the applied stress.
- An adequate huge number cycle of applied stress.

There are lots of ways for conducting a test to measure fatigue as there are ways of applying repeated stresses to a metal. A specimen of rotating beam type is frequently used because it is simplicity. In a fatigue test, the mostly expected output is a number of cycles required to fracture the specimen at a given stress. [17]

The enormous increment life of specimen by which help of continues reduction of stress, hence fatigue data is usually presented by plotting maximum stress (S) against a number of cycles to fracture (N), using a logarithmic scale for the latter variable Fig. 20. [18]

This form of a curve (S-N curve) shown in Fig. 20, is significant because there is a stress below which the specimens do not fracture. This limiting stress is called fatigue limit or endurance limit (σ_e), below which fatigue will never happen. Fatigue limit can be related to the tensile strength of the material, and the ratio of fatigue limit to tensile strength is known as endurance ratio. [17]

Unlike steels, most nonferrous metals do not have a fatigue limit i.e. S-N curve continues to fall steadily with decreasing stress, through at a decreasing rate. Thus, fatigue will ultimately occur regardless of the magnitude of the applied stress.

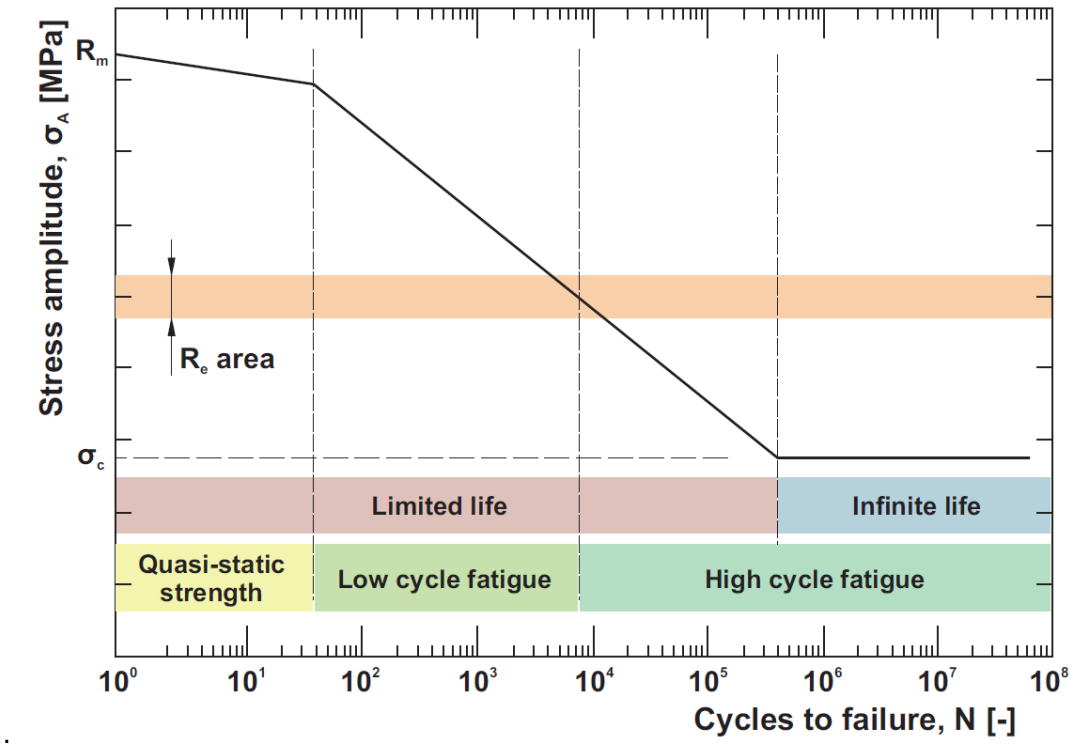

Fig. 20. The typical form of model S-N curve [20]

Fatigue response of these materials is specified for a number of stress cycles, normally 10^7, and is known as fatigue strength. [19]

The typical S-N curve is divided into three zones and all zones depend upon a range of stress. This range of stress decides the number of cycles sustain the materials.

Welds exhibit a significant notch effect. Because of that the cyclic loading method and the expected number of cycles has to be considered during designing and calculating of welds. Depending on the number of cycles in which load welds is recognized:

- **Quasi static strength** - for N number of cycles less than 10^2 cycles.
- **Dynamic load with limited life (low cycle fatigue)** - for N number of cycles in the range of cycles from 10^2 to 5.10^5 cycles.
- **Dynamic load with limited life (high cycle fatigue)** - for N number of cycles in the range of cycles from 5.10^5 to 2.10^6 cycles.
- **Dynamic load with infinite life (high cycle fatigue)** - for N number of cycles more than 2.10^6 cycles.

3.2. Crack initiation and propagation

Based on structural changes that take place during fatigue, fatigue failure process can be as made of following stages: (a) crack initiation (N_i) – includes the early development of fatigue damage that can be removed by suitable thermal anneal (b) slip-band crack growth – involves the deepening of initial crack on planes of high shear stress. This is also known as stage-I crack growth. (c) crack development on planes of high tensile stress (N_f) – involves the growth of a crack in a direction normal to maximum tensile stress, called stage-II crack growth (d) final ductile failure occurs when the crack reaches a size so that the remaining cross-section cannot support the applied load. [17,18]

Total life (N_f) = Crack initiation (N_i) + Crack propagation (N_f)

Fatigue failures normally are found to initiate at a free surface or at internal flaws such as inclusions where the local stress causes some heterogeneous permanent flow leading to the formation of a small crack. The initiation of a fatigue crack does not guide to immediate failure, rather, the crack propagates slowly and discontinuously across the specimen under the inuflence of cyclic stress. The amount of crack motion per cycle depends on the material and the stress level; high stresses favour higher crack growth increments per cycle. The final fracture surface is composed of an area over which there were slow crack propagation and an area where the crack moved quickly. The final fracture can be either ductile or brittle type that observed from the expermiental part [17,18].

In polycrystalline metals, during a fatigue test slip lines visible first on crystal whose slip planes have the highest resolved shear stress. As time goes on and the number of stress cycles increases, the size, and the number of slip bands (clusters of slip lines) increase. As

is shown at Fig. 21 during fatigue occur two types of slips (a) intrusions and (b) extrusions type. [12]

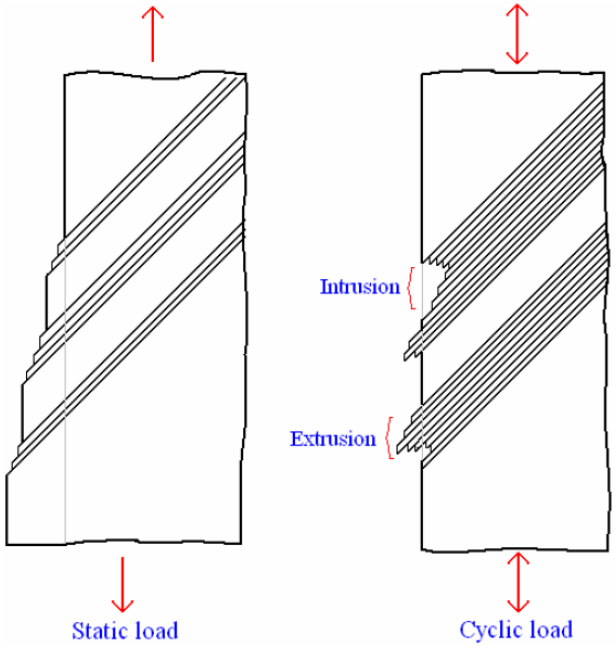

Fig. 21. Comparison of slip bands formed under (a) static loading and (b) cyclic loading.

The crack propagation time from crack begining time, pre-existing flaws or cracks are introduced into a fatigue specimen. Crack propagation rate (da/dN) is studied in terms of increase in crack growth which is directly proportional to the number of applied stress cycles and length of the crack in eq^{n}. (7) i.e

$$\frac{da}{dN} = fn(\sigma, a) = C\sigma_a^m a^n \tag{7}$$

Where:

C	-	a material constant.	(1)
σ_a	-	the alternating stress	(MPa)
a	-	the crack length	(mm)
m	-	2-4. (materials parameter)	(-)
n	-	1-2. (materials parameter)	(-)

It has been found that crack growth rate can be related to stress-intensity factor K of fracture mechanics which itself is a combination of stress and crack length eq^{n}. (8). Thus,

$$\frac{da}{dN} = A(\Delta K)^p \quad (8)$$

where

ΔK	-	stress-intensity factor range.	(-)
A and p	-	constants that are functions of material, environment, frequency, temperature and stress ratio.	(-)

Fatigue data presented in terms of log (crack growth rate) against log (ΔK) is shown in Fig. 22. The curve can be split into three regions. In region-I, which is bound by a threshold value ΔK_{th}, no observable crack growth occurs i.e. crack growth rate is very slow in order of 0.25 nm per cycle or less. The straight line part of the curve in region-II can be represented by the power law (also known as Paris law). [17,8]

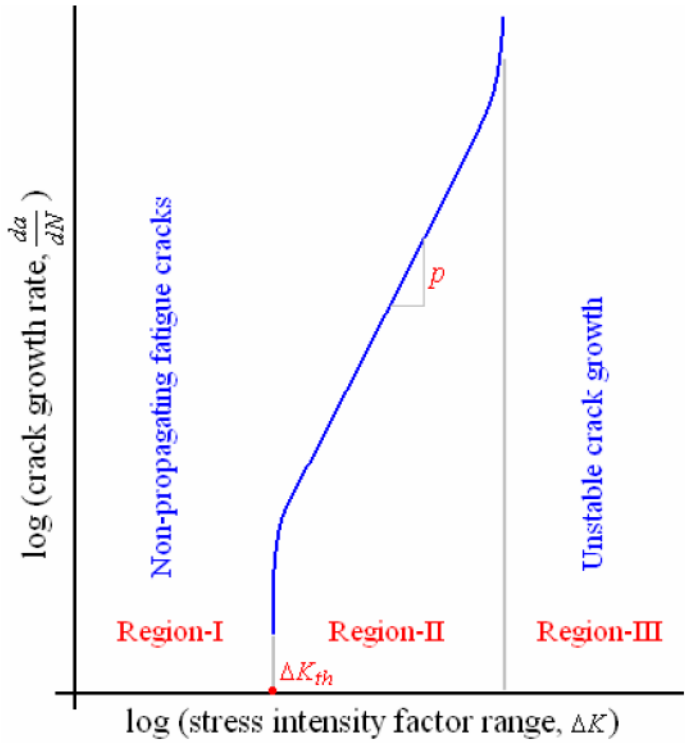

Fig 22. Crack Propagation Curve [17]

4. Experimental

Experimental part is focused on diffusion welding of 10GN2MFA steel and S-N curve measurement. Firstly was meassured of S-N curve of 10GN2MFA steel in a basic state. Then prepared diffusion joints of that material and carried out a dynamic test of diffusion joints. A final result of the experimental part was a comparisson of aplication possibilities diffusion joints during fatigue loads.

4.1. Material (Bainitic steel)

10GN2MFA low-alloy steel came into use for production of the equipment components for nuclear power units with WWER-1000 reactors at the end of 1970. This steel in the specified structures is mainly used for steam generator shells, pressurizes, emergency cooling and protection system capacities, collectors, reactor coolant pipelines, steam separators and some other assemblies were produced from this steel for more powerful (1000 MW) power plants. [25-27] The typical range of the chemical composition of the steel is shown in Table 1.

Table1 - Chemical composition in % for grade 10GN2MFA

Elements	C	Si	Mn	Ni	S	P	Cr	Mo	V	Cu
Min%	0.08	0.17	0.18	1.8	-	-	-	0.4	0.03	-
Max%	0.12	0.37	1.1	2.3	0.02	0.02	0.3	0.7	0.07	0.3

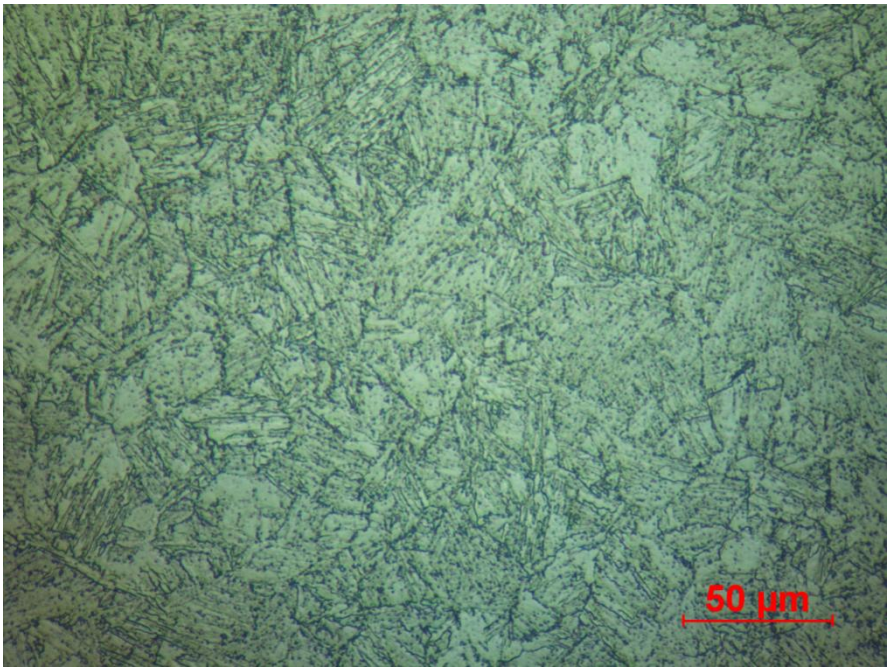

Fig. 23 The microstructure of 10GN2MFA banitic steel

In Fig. 23 there is shown microstructure of 10GN2MFA steel in the basic state. It is contain pearlite structure along with tempered bainitic structure. Pearlite with banitic gives optimal mechanical properties like Table 2.

This steel is characterized by quite high heat-resistance by high strength and plasticity due to optimal alloying of the solid solution and to high deep-hardening properties. This type of low alloy steel called bainitic steel. The significantly modernized technology of the 10GN2MFA steel production (electric arc furnace, open-hearth process, refining in ladle furnace, vacuum degassing, bottom pouring under argon protection into the mould). All this production process allows to enhancing considerably micro-cleanliness and chemical composition of the steel but steel that is produced by such method is not sensitive to stress corrosion cracking. This steel is focused on optimization of these fundamental utility properties by inter critical annealing heat treatment process under investigation to increase toughness and critical temperature of brittleness. Strength limits on the low alloy steel of different elements do not have sufficiently justified quantitative criteria which could be used for evaluating the effect of the chemical composition of steel on the stress corrosion cracking, in addition, elements are the formation of fine grain structure and homogenous. The steel 10GN2MFA can be welded by many common welding methods. For the Czech Republic, The company Vítkovice Heavy Machinery as. used for the production of collectors for the Temelin nuclear power plant an electric arc furnace, followed by the refining of the oil in the pan, vacuum degassing and pouring under the protective atmosphere of argon. The resulting steel contains less than 0.010 % P and 0.005 % S, meeting the prescribed limits for the Czech Republic.

The heating temperature has an adverse effect on the mechanical properties because it occurs to the growth of austenitic grain and to the formation of metastable structures. After austenitisation at T =1 200 ℃ is an isothermal delay between 600 and 700 ℃ in the furnace for 2 to 50 hrs. method. [28] The measured mechanical properties of the 10GN2MFA steel in the basic state are shown in Table 2. According to these mechanical properties set up all stress amplitudes for the fatigue testing. Fatigue all stress amplitudes form higher one to lower one between the range of yield strength.

Table 2- Basic mechanical properties of 10GN2MFA steel [34]

$R_{p0.2}$ [MPa]	R_m [MPa]	A_g [%]	Z [%]	KVC [J.cm2]	T [℃]
534	629	24	74	59	20

The CCT diagrams can be used to predict the microstructures, and thus the properties, which would result from a specific cooling rate, whether due to fabrication, thermal processing, or welding and as such are important for engineers and designers in the development, processing.

In TUL was measured CCT diagram of 10GN2MFA steel for cooling rates from 85 till the 0.05 °C.S^{-1}. This information is very important for the design of the right cooling rate during diffusion welding of 10GN2MFA steel. It is important to receive the same or very similar structure and mechanical properties which are in a basic materials. In Fig. 24 there is CCT diagram of 10GN2MFA steel measured with help of two different dilatometers (Gleeble 3500 and Bahr 805L) with different physical heating methods.

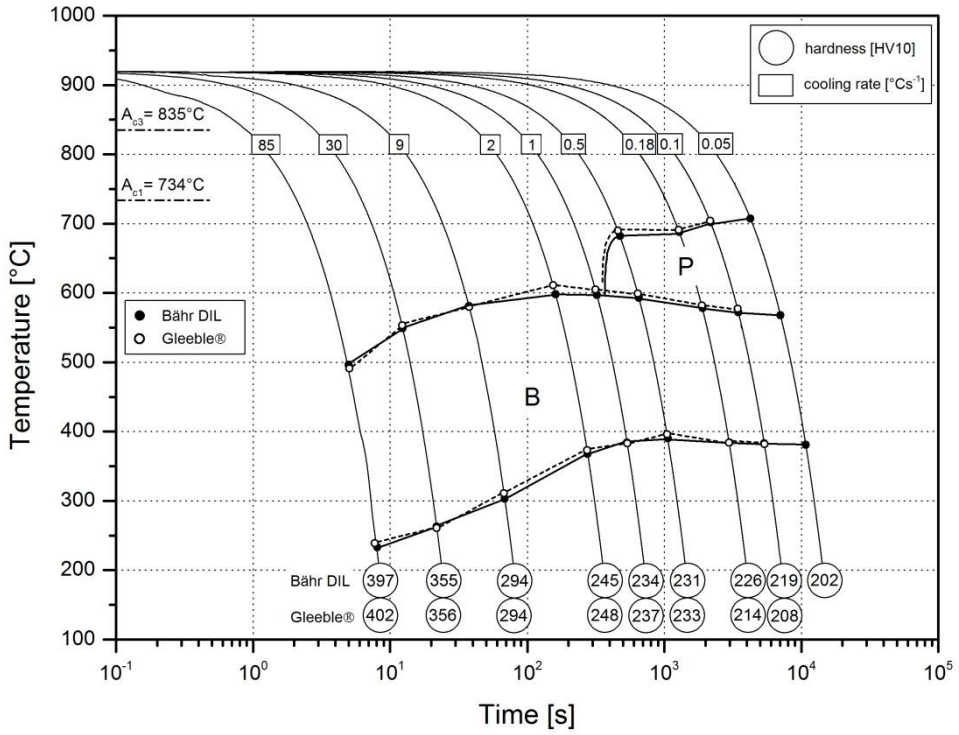

Fig. 24 Cooling curve at Gleeble device [34]

4.2. Fatigue properties of 10GN2MFA steel

Fatigue properties of the bainitic steel materials belong to among the very important material parameters, mainly because they are very closely related to the total fatigue life of the part. Knowledge of the boundary between limited and endurance life represents a truly most important fatigue parameter. These tests were performed under σ_{mean}= 0. As a major aim, there was a determination of so called S-N curves (stress vs. number of cycles in log scale) and this curves given basic pre deformation 10GN2MFA steel on the cyclic load. From the sample preparations point of view, there were very important to ensure that the final

fracture will be in the working area of the sample. In Fig. 25 there is shown a 3D model of fatigue sample and also drawing of that sample with all constrains. All specimens were tested under cyclic axial loading at a nominal stress range, $\Delta\sigma_{axial}$.

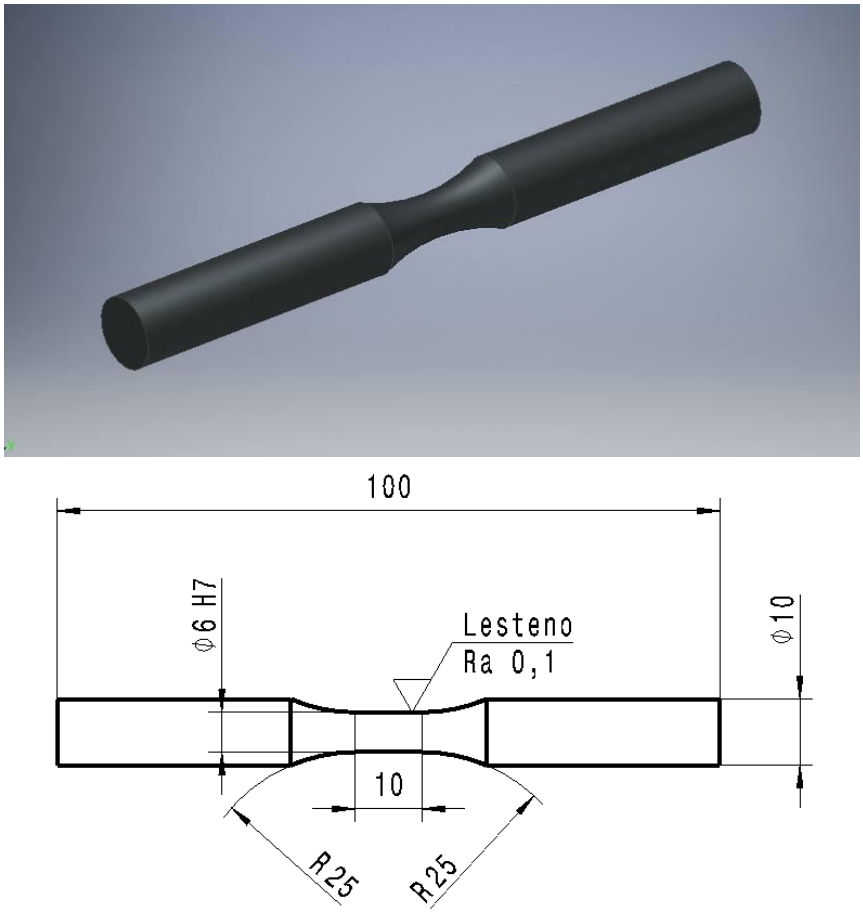

Fig. 25 Schematic 3D sample and sample diagram

The own testing was performed on the loading machine FU-O-160-1600-V2 (company INOVA - see in Fig.26). Required stress amplitude σ_A [MPa] was adjusted via the force amplitude F (N) and the relevant cross-section S (mm^2). After that was sample loaded up to its failure. All acquired result is shown in Table 3. The load group A started with 60% yield strength of 10GN2MFA steel. Endurance limit showed upto the load group D in Table.3. This was the safe group of load where fracture did not happen 10GN2MFA steel. The load group from E to J were placed in the limited life area of S-N curve shown in Fig. 27. Load groups E and F are on a boundary between high and low cycles fatigue and the rest samples are in low cycles fatigue.

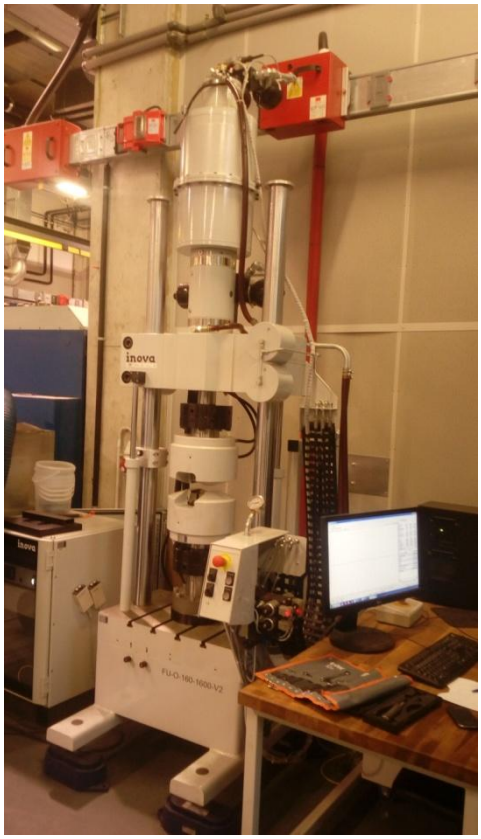

Fig. 26 Fatigue test machine

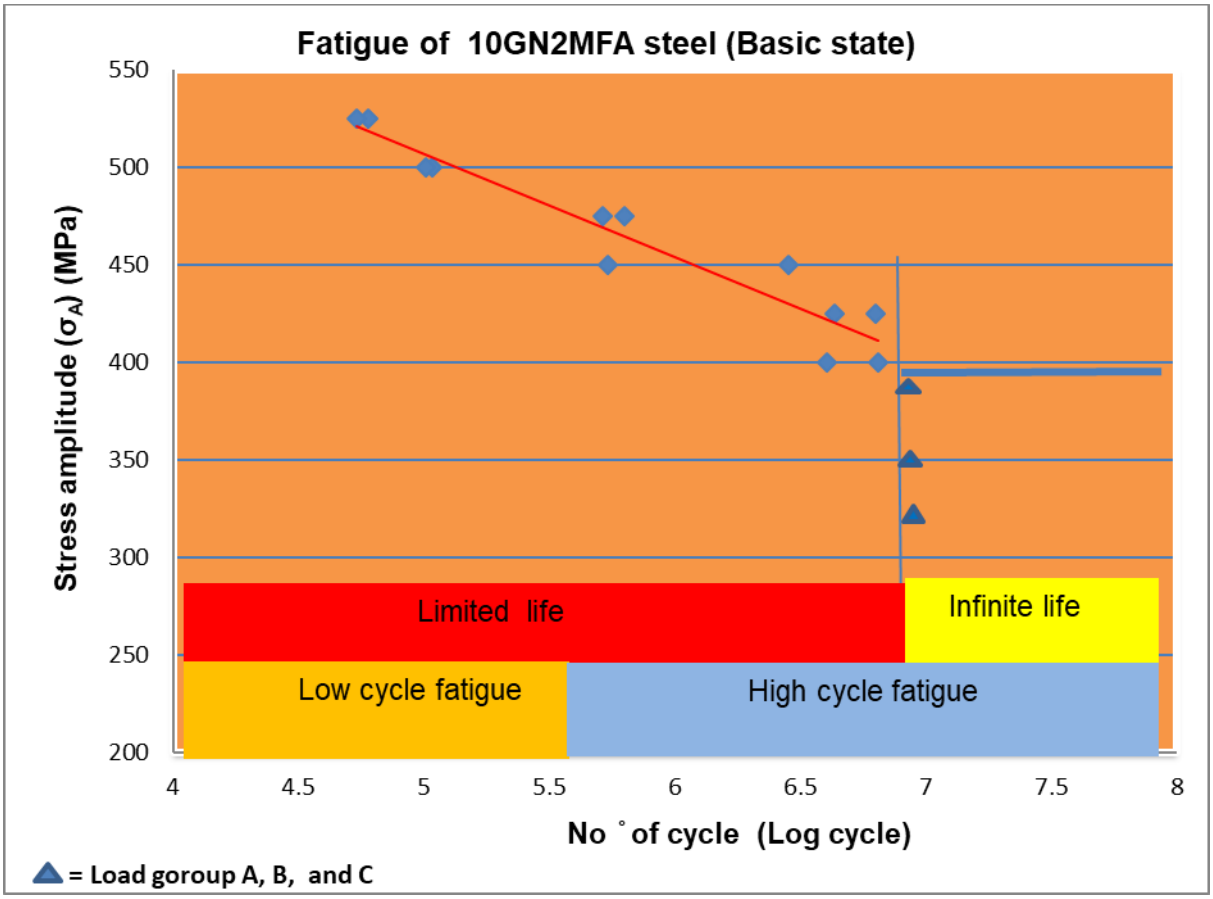

Fig. 27. S-N Curve of basic 10GN2MFA steel

Table 3: Comparing stress amplitude with No˚of the cycle (Basic state)

Load group	Stress amplitude (MPa)	Mean stress	$\Delta\sigma_{axial}$ (MPa)	Frequency (Hz)	No of cycle	Crack (Yes/ No)
A	325	0	650	40	$> 10^7$	No
B	350	0	700	40	$> 10^7$	No
C	385	0	770	40	$> 10^7$	No
D	390	0	780	40	$> 10^7$	No
E	400	0	800	40	5249534	Yes
F	425	0	850	40	5307645	Yes
G	450	0	900	40	1691363	Yes
H	475	0	950	40	572498	Yes
I	500	0	1000	40	104981	Yes
J	525	0	1050	40	56882	Yes

All result from Table 3 were putting into the graph Fig. 27. It explains stress amplitude vs number of cycles in log scale was used as a linear approximation. In the graph, there is shown limited life (low and high cycle fatigue) with help of red line and infinite life with help of blue line. Infinite life was set (for the basic state of 10GN2MFA steel) at a load level of 390MPa.

4.3. Experimental creation of difusion welds

The Gleeble can be used for many different stress-temperature types of simulation. At the Technical University of Liberec the device has been used predominantly for academic activity, however, the device is extensively applicable to the industrial sector as well. In the field of energy, the instrument has already been used to test the creep resistant material during multi-layer welding, cyclic thermal stressing of materials used for steam turbines, or plastic deformations of austenitic materials. The Gleeble device was used for experimental part of this diploma thesis. 12 number of samples were prepared by the diffusion welding with same parameters (heating rate, cooling rate, holding temperature and holding time, pressure, vacuum etc.).

4.3.1. Design of a diffusion welding experiment

Before the welding process, there are lots of pre processing. These pre-processing process affects on both the welding parameters as well as the Gleeble machine. The Gleeble machine was used for diffusion welding. This process is very sensitive to the right welding parameters.

For experiment was used 10GN2MFA steel samples dimensions 50 mm length and 10 mm diameter. Each sample was necessary to attach by thermocouple on one of the welded parts to sensing and controlling the temperature of the connected parts throughout heating, holding and cooling process. The thermocouples were welded using a condenser welding machine Fig. 28. The 31 volt recommended for thermocouple joints on this material. Place where the control thermocouple as close as possible to the point of connection (up to approx.0.5-0.7 mm from the joint edge) in Fig. 29. On the said location was subsequently condensed by welding the individual parts of the thermocouple type K. These were 0.25 mm diameter wires composed of Ni-Cr (+) and Ni-Al (-).

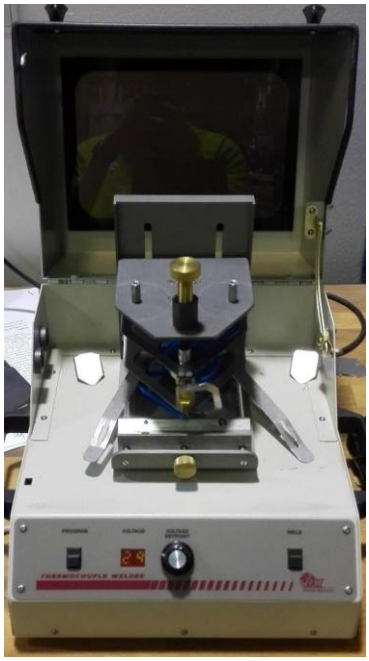

Fig. 28 Thermocouple welding machine

Fig. 29 Sample with thermocouple

It's about the most widely used type of thermocouple used in vacuum tests, designed for temperatures not exceeding 1250 °C. To avoid touching the two stripped conductive parts thermocouple was used a ceramic protector, which fulfills the function of an insulator.
After a thorough visual inspection, both 10GN2MFA samples (with or without a thermocouple) was embedded in full-contact copper jaws and clamped into the vacuum clamping device of the Gleeble apparatus chamber, shown in Fig. 30. Contact area on both parts of samples was cleaned with acetone because of each mess decrease strength of final diffusion joint.

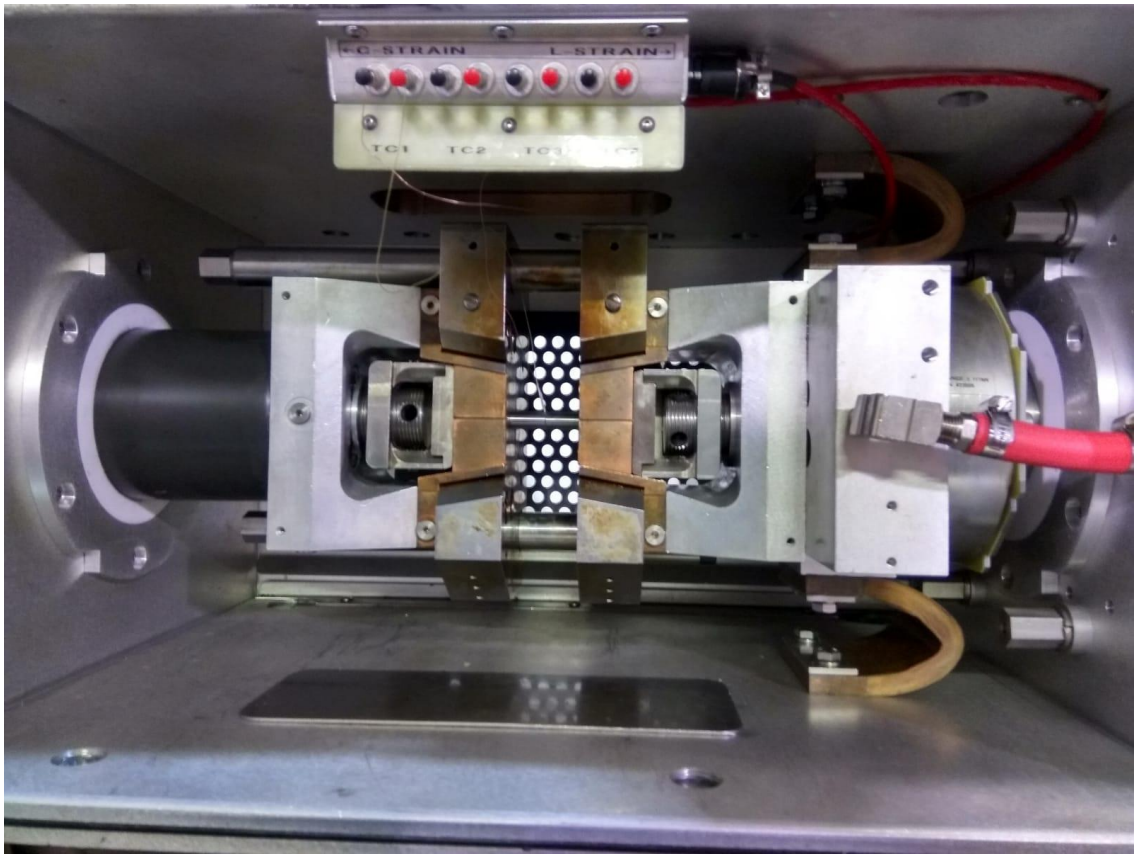

Fig. 30 Sample in welding chamber before the weld

All experiments were performed with full contact copper jaws which are used due to a steeper temperature gradient, and hence, smaller heat-affected areas at the interface of samples. When inserting jaws into the Gleeble vacuum chamber was necessary for the conact area of the rollers to be the same plane with the end plane of the clamping jaws. At the same time, clamping device of the jaw the specimen is spanned by screw spacers to make good contact, and transfer of heat energy between the clamping device, the jaws and the sample, and also to the sample was fixed with sufficient force and did not move during the experiment. This could affect the compressive force of the experiment. There after, the

samples were mechanically brought together so that their faces touched, but at the same time, no force greater than 0.1 KN was derived. The chamber was then vacuumed and after reaching a vacuum value of 1.3 x10^{-3} Torr experimental welding could be started.

This diploma work follows the bachelor thesis of Jakub Vrba [34], where the process parameters of diffusion welding were optimized. According to the bachelor thesis, the best process parameters of diffusion welding for 10GN2MFA steel are temperature 1125 °C, holding time 20 min. and pressure 7,1 MPa. Cooling rate after the holding time heavily affects the microstructure, ductility properties, and hardness as well. In Table 4. represents 70% higher ductility in the cooling rate of 0.1 °C.S^{-1} compared to the cooling rate 10 °C.S^{-1}. The difference in the ductility such a high range due to the microstructure phase. These process, parameters were achieved the same ultimate stress joint as achieved for basic material.

Table 4- Mechanical properties according to different cooling rate [34]

Sample with different cooling with force	D[mm]	R_p 0.2 [MPa]	R_m [MPa]	A_g [%]	A_{20} [%]
T1125_F1,2_t20_cool0,1	8.02	490.5	687.7	7.52	19.88
T1200_F0,6_t20_cool10	8.03	812.3	891.2	0.39	0.43
T1050_F1,2_t20_cool10	7.93	727.1	834.0	0.75	1.23

The Gleeble is driven by a QuikSim2 system that lets set up all required parameters for a run successful test. This is simply a table system in which is possible set the time and range of the appropriate variable, or several variables, to define the rate and intensity of the changes in the sample being tested. Except defining the sequence of the test method can be defined in QuikSim2 quantities, including the frequency of their recording Fig. 31 shows the program created for diffusion welding of the specimen at the first 5 seconds has been set to zero pressure force and temperature of 20 °C due to the correct start of the system. During the next 5 seconds, the temperature rose slightly to 25 °C and a pressure force of 0.8 KN began to occur, which remains the same until the end of the experiment.

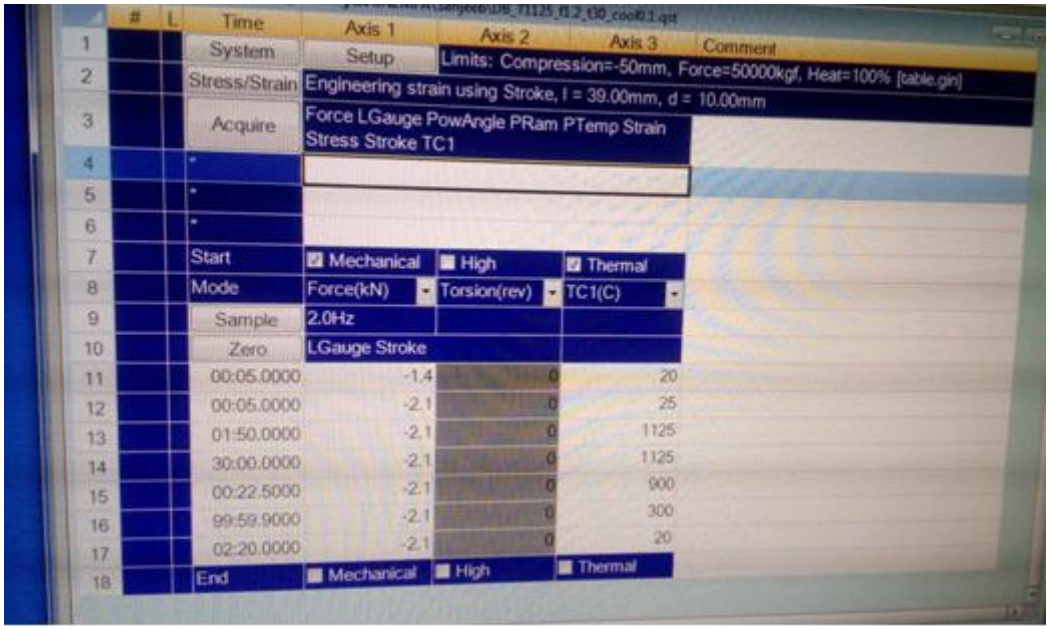

Fig. 31 Quiksim software with all parameters

An increase of temperature to 1125 °C was reached at 1.5 mins., corresponding to a heating rate of 10 °C.S^{-1} and holds this temperature for 30 mins.. Then cooling followed by 900 °C for 22.5 sec., this corresponds to cooling rates of 10 °C.S^{-1}. The cooling time between 900 °C and 300 °C was set to 100 mins, this corresponds to cooling rate of 0.1 °C.S^{-1}. The last two minutes and 20 sec. served to cool the samples at the original 20 °C, which corresponds to a cooling rate of 2 °C.S^{-1} (free cooling). The cooling the process was chosen based on the knowledge of the CCT diagram so as to achieved pearlite and bainitic structure. The frequency of the sensing of the quantities was selected 2 Hz. In this case, this was specifically a recording force (Force), the measurement of the elongation (size of plastic deformation) using (LGauge), the current transformer power (PowAngle), programmed movement of stroke (PRam), programmed temperature PTemp, next Strain, Stress and real temperature from the control thermocouple (TC1). When welding was started a lot of heat was produced near the joint area in Fig. 32 and 33 (More close look), because heat and pressure concentrated in the joint area to begin weld. Heat affected zone (HAZ) nearly 6 to 8 mm from the joint area. HAZ was directly or indirectly affects the quality of the joint area and depends upon which technology uses for welding. However, lower HAZ is directly proportional to the good quality sample joint

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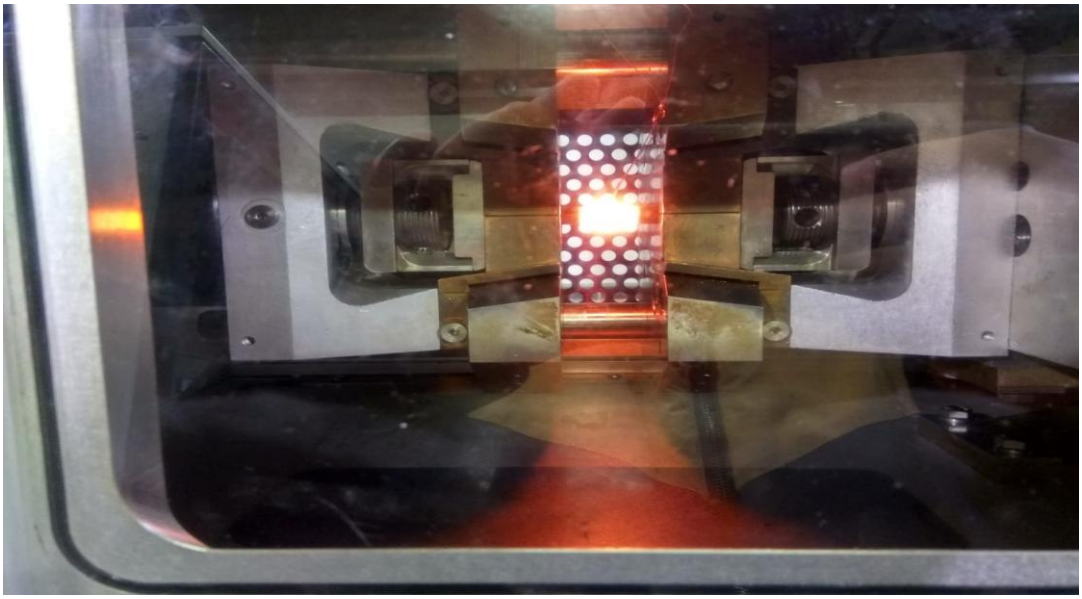


Fig. 32 Sample while welding

Fig 33 Near image welding zone

Although the 10GN2MFA diffusion welding experiment is fully in place automatically, however, it was important to personally supervise the process. Even with seemingly the successful joint of the thermocouples on specimen but it may be dropped during the very high-temperature welding practice. Because it exposed to the high-temperature environment. When the samples are heated to a predetermined welding temperature, the value varies considerably with current transformer power (PowAngle). The reason is a pulsed flow of electric current at the interface between the materials caused inadequate contact on the transition areas. The temperature raises slightly plastic deformation on the contact surfaces and partial diffusion connection in deformed places. This is reduced the

pulsation until it becomes even increasing the required performance. The transformer power is already at the exposure temperatures almost constant. [28,34]

4.3.2. Evaluation of an experiment

After the design the experiment, it was necessary to know that all parameters were suitable to the good quality of welding. Quality of joint can be found by the metallographic process in Figs. 34 and 35**.** The goal of this part of work was to verify the chosen process parameters generate joint of corresponding quality. It means whether the interface of the joint is without any defect. Thank this is possible confirmed the process parameters or suggest their change.

The result of the metallographic evaluation is shown in Fig. 34. Samples were prepared by the standard way (grinding and polishing). For highlighting of the structure was used etching with 3% Nital (3% HNO_3 in distilled water). All metallographically prepared samples were evaluated by optical microscopy. The middle of the sample was without defects but close to the surface of the sample, there were indicated some pores. This indicates not enough time for full diffusion in all cross section of the sample. This could have a high impact towards the mechanical properties of joint especially fatigue. So it was a reason for elongation of holding time from 20 to 30 minutes at 1125℃.

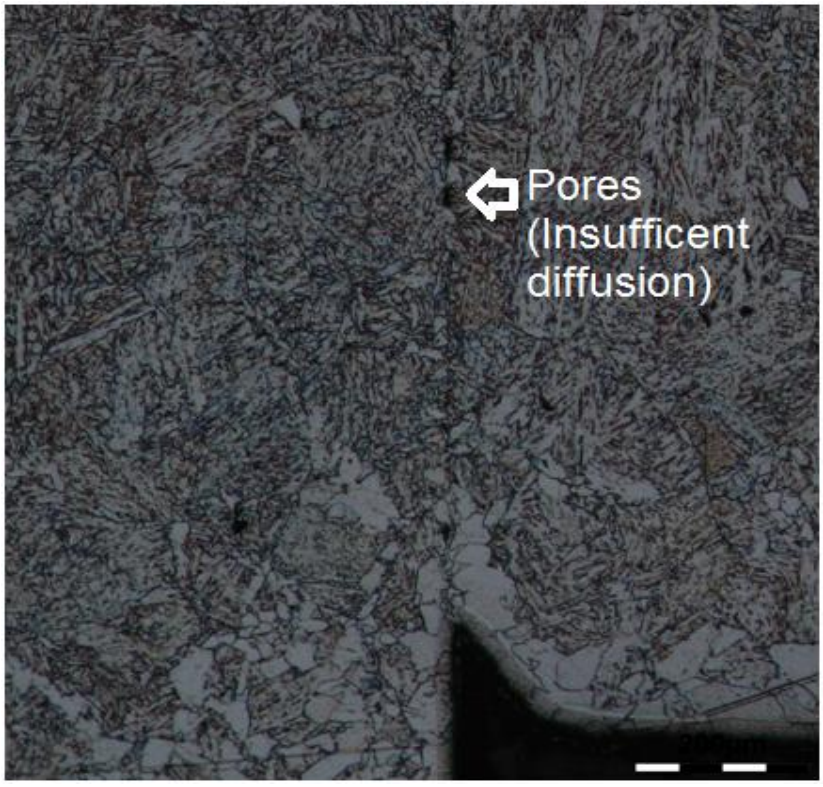


Fig.34.Microstructure 30mins holding time at 1125℃

The result of using longer holding time is shown in Fig. 35. This joint was adequate for the further mechanical test.

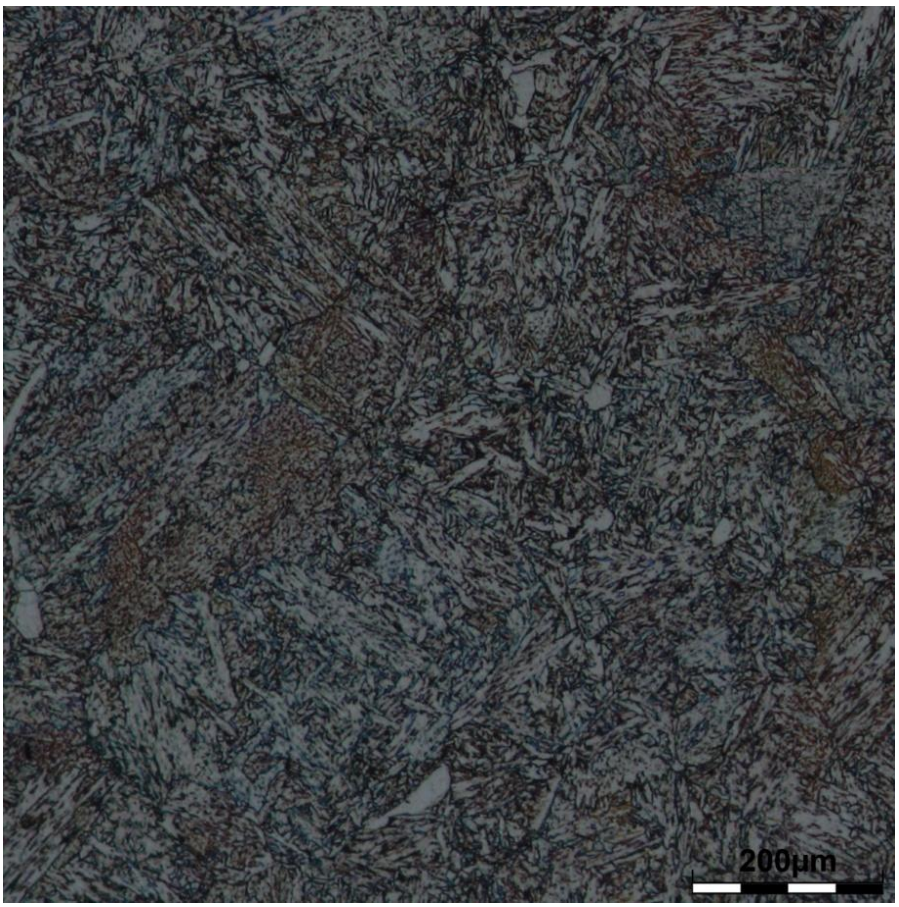


Fig.35.Microstructure 30mins holding time at temp. 1125℃

On sample welded with force 0.8 KN, temperature 1125℃, holding time 30 mins and cooling rate 0.1 °C.S^{-1} was measured hardness HV10 and microhardness HV0.5. The graph Fig. 36 shows that the hardness HV10 at the welding zone and heat affect zone slightly less compare to the base metal. Hardness has decreased approximately about 12 HV. The distance between the individual impacts indenter was 1 mm. Heat affected zone is similar 15 mm from the welding site.

The graph of micro harness HV0.5 is shown in Fig. 37. The distance between the individual impacts was 0.5 mm. The micro hardness map indicates that the hardness in the Fig. 35 of the steel is the lowest at the interface area. The interface area joint as well as HAZ exhibits a hardness of 220 to 230HV. On other hands the both Figs. 36 and 37 show hardness approx. 245 to 255 HV far away from the joint area.

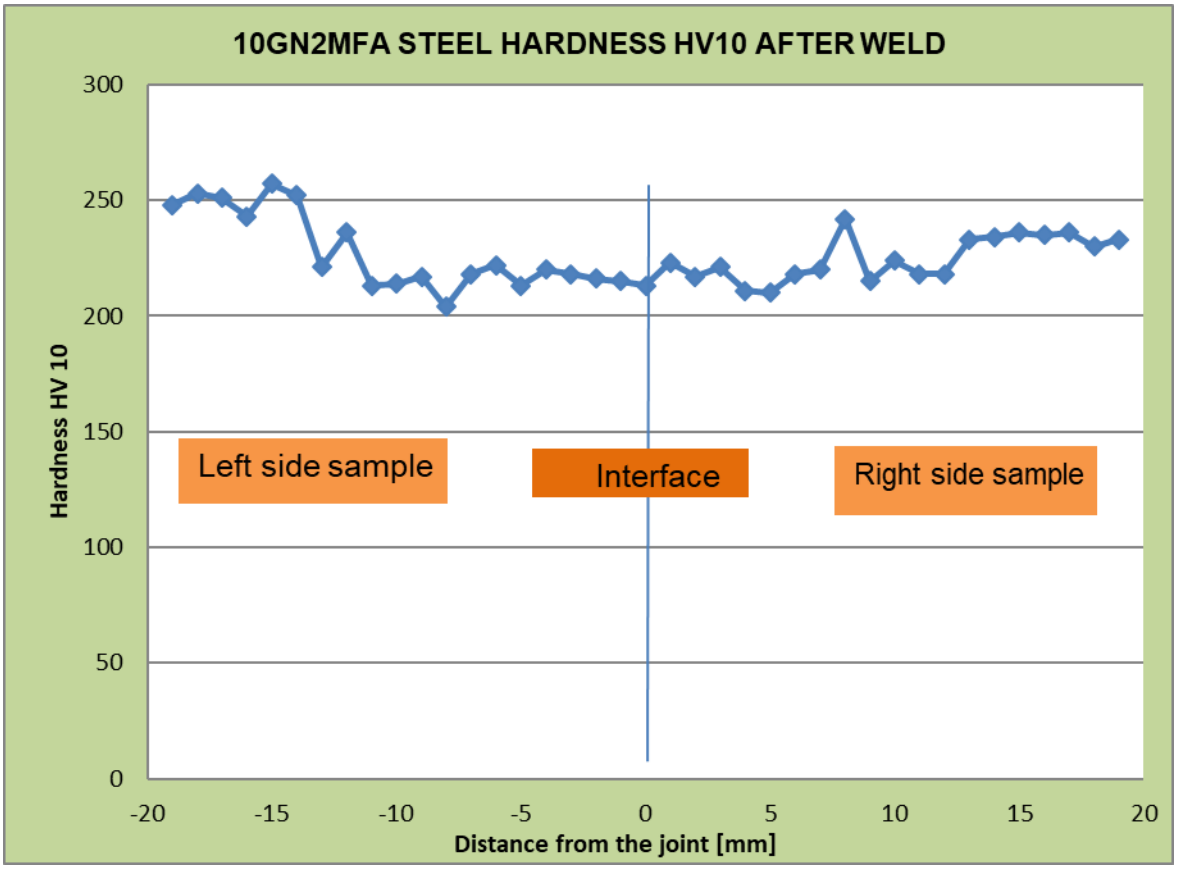


Fig. 36 The hardness of 10GN2MFA steel after the weld

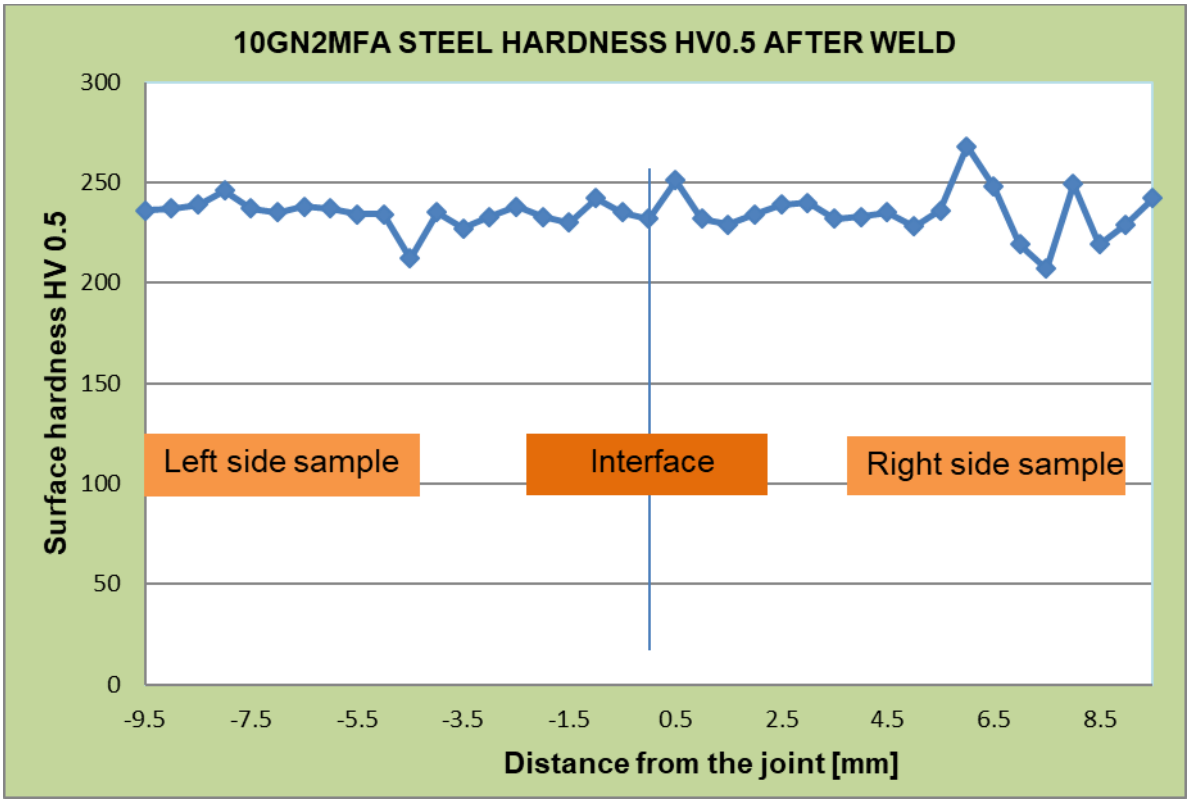


Fig. 37 Surface hardness 10GN2MFA steel after the weld

In addition to the hardness and microhardness measurement and the evaluation of the specimen by optical microscopy, were diffusion welded samples monitored in terms of deformation during the holding time on working temperature. In Fig. 38 there is shown deformation of sample number 5. The deformation flow was under holding temperature 30 mins. Fig. 39 displays the image of the sample number 5 after welding and on removal from the Gleeble device.

All the parameters evaluated have shown that such process parameters can be used to create a good quality of joint. This was also confirmed by the tensile test, where a yield strength of R_e = 496 MPa, an ultimate strength R_m = 694 MPa, elongation A_g = 7.48 % and an elongation A_{20} = 19.34 % were obtained at the diffusion joint.

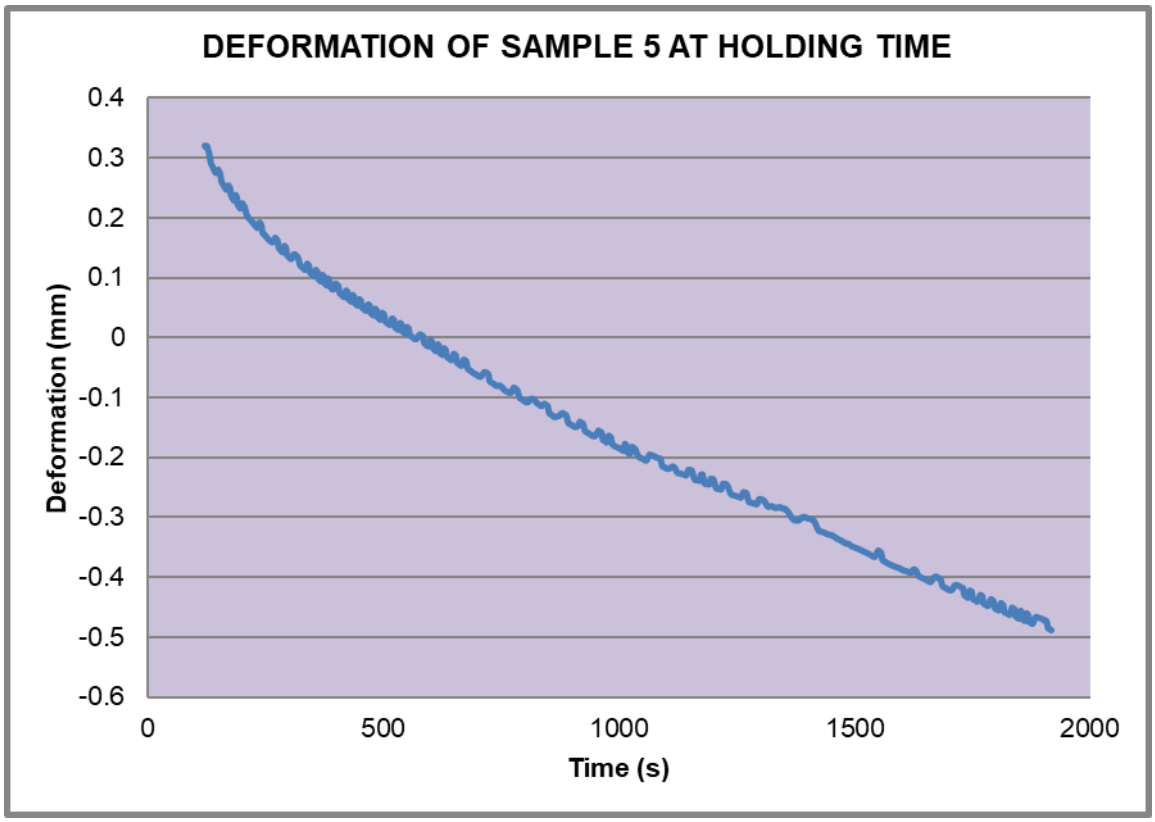


Fig. 38 Deformation sample 5 at the holding time

Fig. 39 Diffusion welded Sample 5 under the mentioned parameter

Therefore, another 9 samples were welded under the same conditions. These samples were machined to suitable shape and used to the creation of the S-N curve. The observed parameters of diffusion welded samples are given in the attachment.

4.4. Experimental S-N curve measurement on diffusion welds

Diffusion welds samples were prepared for the fatigue test by machining. A lot of parameter importance for fatigue test has a surface roughness. Surface roughness highly influences the fatigue mechanical properties. In Fig. 40 there are shown two images (A) and (B), before and after the polishing. Here the differnence could observed between roughness according to shinining.

The software was used for the input all parameters of the fatigue test into the machine is shown in Fig. 41. Input parameters were frequency, stress amplitude (given by force in KN) and mean stress. Frequency is really important for each fatigue test. During the test it was important to observed surface area behavior. According to high frequency 60 Hz during stress amplitude 465 MPa, the high deformations produced at high heat which leads to working area of the sample to the temperature about 350 °C (blue color on a sample's surface).

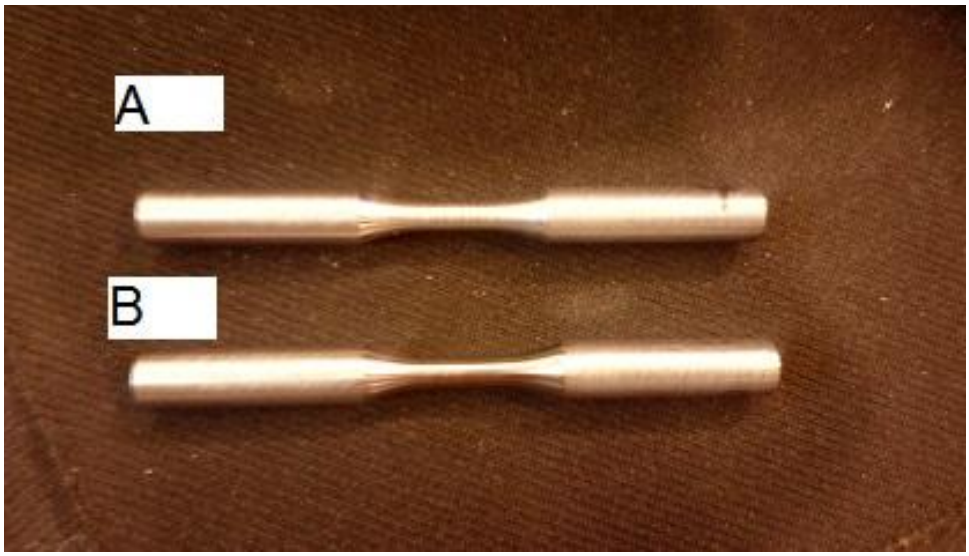


Fig. 40 Sample difference beetwen polishing (A) Before polishing (B) After polishing

Samples were fitted into the clamps of the fatigue machine (Fig.43). Started the testing according to given parameter and simultaneously screen look like Fig.42. Curves are running from highest point of stress amplitude to lowest point and the number of cycles are

counted till fracture. When driving system stopped the test and number of cycles stay the same. In Fig. 44 there is shown the window of software after the fracture.

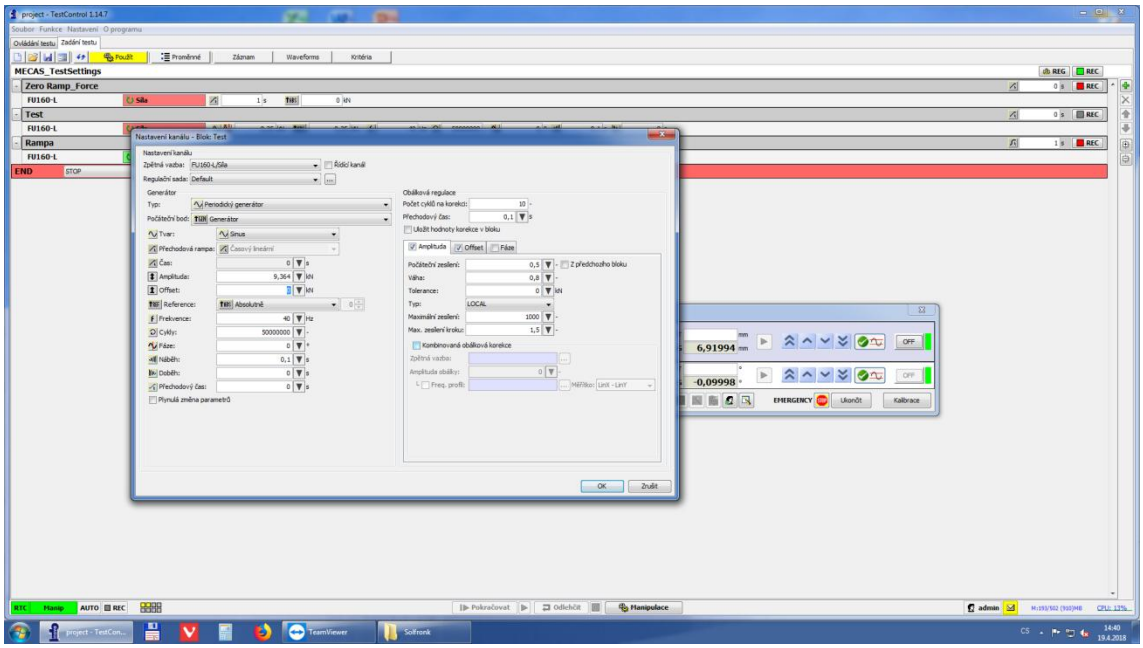


Fig. 41 Software for parameter setting before test start

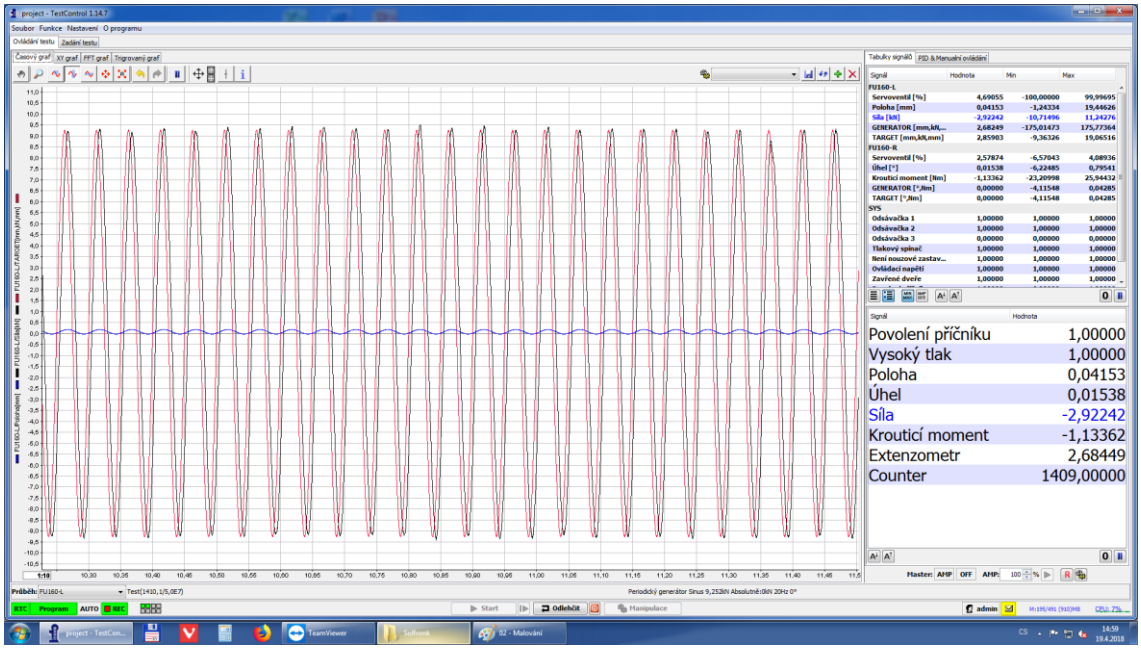


Fig. 42 Software during the testing under the frequency 40HZ

Fig. 43 Sample at fatgiue testing machine under the clamp

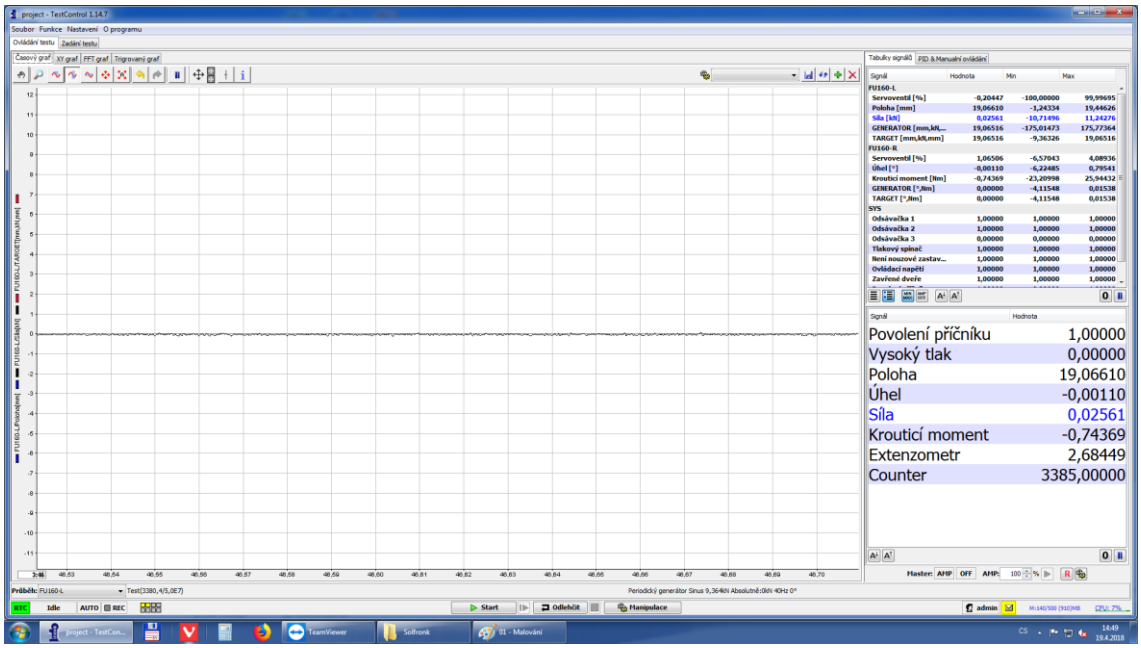


Fig. 44 Software after sample fracture

All the load groups and frequency was highly important for number of the cycles. In the Table 5 there are mentioned all the load groups along with the count of the cycles. Stress amplitude start from the 280 MPa as a lower one and is finished with 475 MPa as a higher stress amplitude. Eight load groups had been used for getting the S-N curve (Fig. 45) of the

diffusion welded samples. When stress level more than 300 MPa, fracture happened in the joint area. Below the 300 MPa stress level, samples were running in the area of infinite life. The fracture may arise in an area of diffusion weld or outside this area. If fracture arises in interface (place of joint) contact of both materials, it means that joint has not the good quality for used load stress. If fracture arises outside interface, it means that joint has the good quality and the fracture happened because of impurities or foreign particles in the steel.

Table 5 represents the values gained during fatigue test under different load stress level. Stress amplitude 308 MPa was sustaining about 413269 number of the cycles but stress increased 42 MPa equal to level 350 MPa means decreasing the cycles number to 273544. Number of cycles 81237 sustained in stress 400 MPa and for stress 475 MPa samples are contiuned to withstand up to cycles number 6891.

The S-N curve Fig. 45 represented y-axis stress amplitude (σ_A) and x-axis number of the cycle (in log scale).This curve divided into the 4 groups. The groups are limited life, infinite life, low cycle fatigue and high cycle fatigue. Infinite life stress below the endurance limit in Fig. 45 shown with help of a blue line. The stress level of blue line was 298 MPa. The limited life is characterized by fracture and it starts under the number of cycle 10^7. Here fracture can be seen from the stress level 308 MPa in Table 5 and in graph mention as a limited life.

Table 5 - Comaprison of Stress amplitude and No˚of cycles after diffusion weld

Load group	Stress amplitude (MPa)	Mean stress	$\Delta\sigma_{axial}$ (MPa)	Frequency (Hz)	No˚of cycle	Crack (Yes/ No)	Crack in place of joint
A	280	0	560	40	$> 10^7$	No	No
A1	280	0	560	40	9 320 475	Yes	No
B	298,5	0	595	40	$> 10^7$	No	No
C	308	0	616	40	413269	Yes	Yes
D	315	0	630	40	472928	Yes	Yes
E	332,5	0	665	40	425504	Yes	Yes
F	350	0	700	40	273544	Yes	Yes
G	400	0	800	40	81237	Yes	Yes
H	475	0	950	40	6 891	Yes	Yes

A limited life divided into two parts low cycle and high cycle fatigue including infinite life. The boundary between low and high cycle fatigue is different and according to the available literary sources and moves in the range from 2.10^5 to 5.10^5 of cycles.

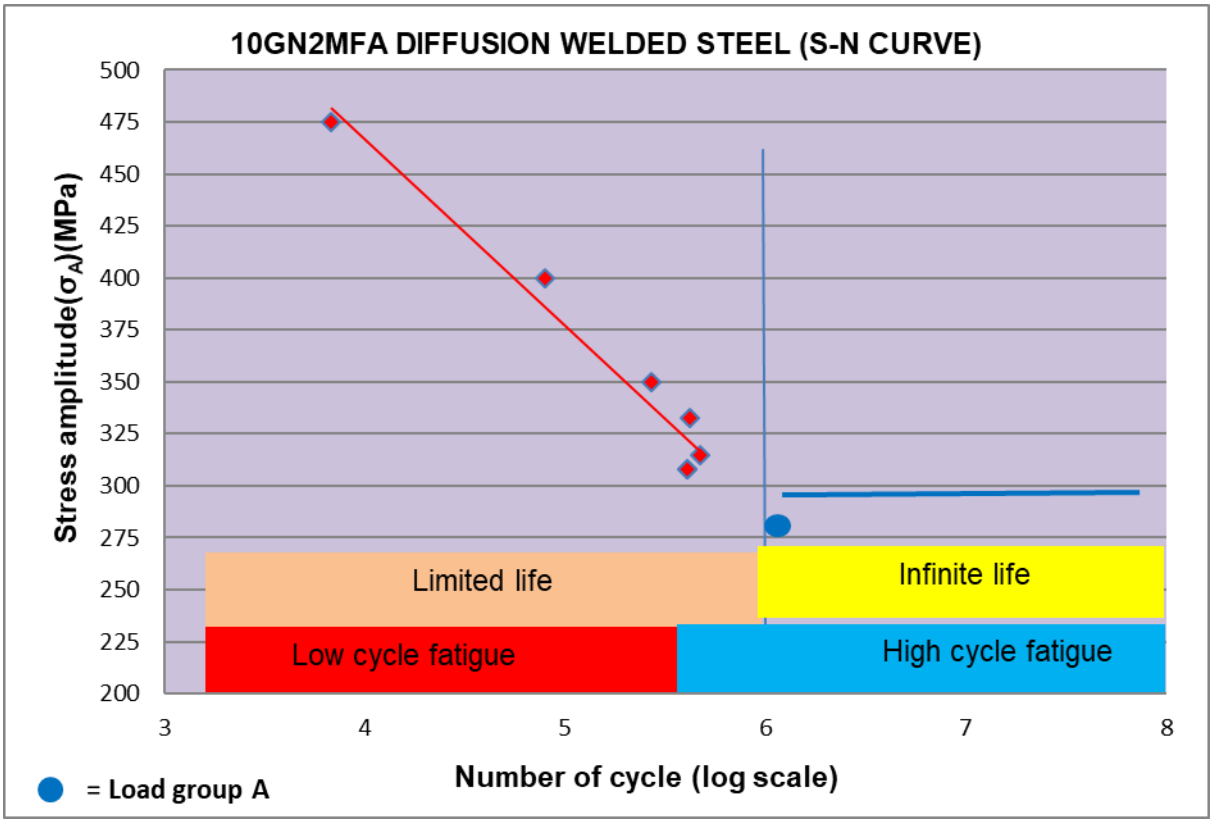


Fig. 45 S-N curve for welded steel of 10GN2MFA

5. Result and discussion

The mechanical properties of the joint obtained by diffusion welding are strongly dependent on the loading method based on the results of the experimental data. While the mechanical properties of the diffusion joint (from bainitic steel 10GN2MFA) are identical with those of the base material under static loading, the dynamic differences between the diffusion joint and the base material are evident in dynamic loading. The metallographic evaluation of the diffusion joints generated at 30 mins at 1125 °C and 0.8 KN load showed no discontinuity at the joint site. This was confirmed by the static pull test. However, at higher stress levels of cyclic loading, a fault occurred at the point of diffusion. This probably means that diffusion has not entirely occurred at the atomic level and there are still discontinuities at the point of coupling, but these are not visible when magnifying the corresponding optical microscopy. At lower stress levels (Load group A1, Table 4), these discontinuities are no longer reflected and the sample breaks out of the diffusion area.

The cooling rate selected by the CCT diagram was correct and the same structure as the base material was obtained, which confirmed the hardness and microhardness measurement in the weld, HAZ, and base material range.

Fig.46 compared the S-N curves for basic steel and welded 10GN2MFA samples. For basic steel shows infinite life on load level 390 MPa and for welded samples, the infinite life is on load level 298 MPa. The difference is nearly 24%. Also in the area of limited life are difference very similar. The only guideline is a bit steeper.

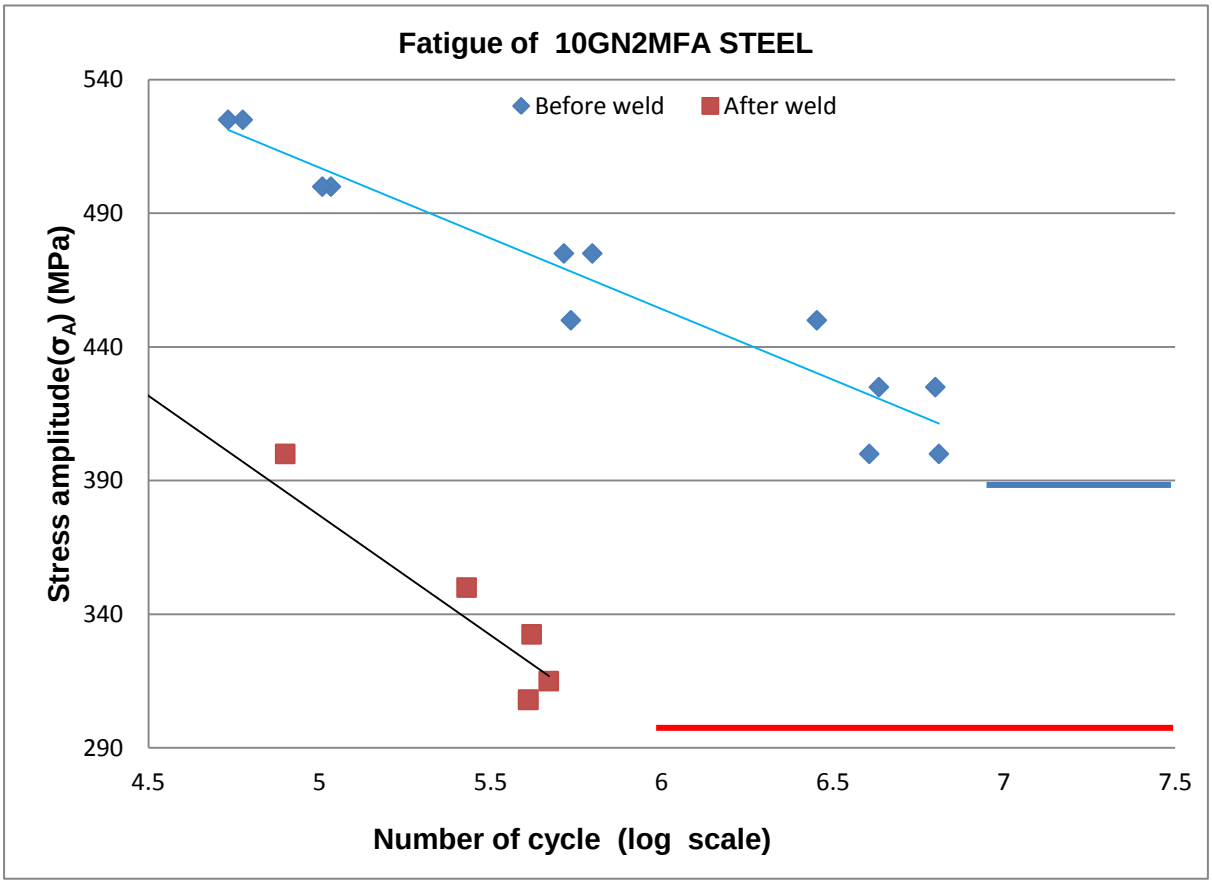


Fig. 46 Comparing S-N curve before and after welding

Table 6 represents the value of stress amplitude and their respective number of cycles. In the stress amplitude, 400 MPa the diffusion-welded samples survives up to 5168297 less number of cycles to compare with the basic material. At 475 MPa, the welded sample behaves very fragile. These samples could not be survived at the 10^4 cycles and the fracture happened 6891 number of cycles. On the other hand sample without welding can be stand 572498 number of cycles on the same amount of stress amplitude.

Table 6 - Comparing stress amplitude and cycles between basic and welding sample

Stress amplitude (MPa)	Number of cycles		Crack (Yes/No)	
	Before welding	After welding	Before welding	After welding
350	$> 10^7$	273544	No	yes
400	5249534	81237	yes	yes
475	572498	6 891	yes	yes

6. Conclusion

The main task of diploma thesis was to judge the influence of diffusion welding of the 10GN2MFA steel on mechanical properties during fatigue loading. The lessons learned can be summarized in several successive points.

1. The microstructural examination of the weld cross-sections revealed that the welds were free of any defects such as porosity, concavity, voids, inclusions or misalignments. This indicated that the welding parameters used for they are appropriate to obtain sound welds.
2. Deformations during the diffusion welding are small without affecting the resulting shape of the final sample. However, they could be smaller, with a slight reduction of the compressive force by about 5%.
3. Cooling rate 0.1℃/S highly important for receiving the same structure as the basic material. In a case of the different structure of the basic material is necessary to change cooling rate according to CCT diagram.
4. Hardness and microhardness measurement help to evaluate if and how big influence has welding cycle to final structure and also it is the well-chosen cooling rate.
5. The selected welding parameters are perfectly suitable for statically loaded joints. In the case of dynamic loading, it is likely to be necessary to extend the holding time on welding temperature to remove minor discontinuities in atomic level.
6. Frequency is highly important parameter during fatigue testing. To high frequency (at high deformations) can generate heat in a sample and increase sample's temperature.
7. Determined and experimentally tested diffusion welding parameters allow dynamic loading of 10GN2MFA steel at stress amplitude of 298 MPa. This is about 24% less than the basic 10GN2MFA steel.

This topic can continue with the way of crack analysis. It going to help us in th field of origin of failure, primary and secondary crack, failure mode and mechanism, the orientation of stress and surface analysis.

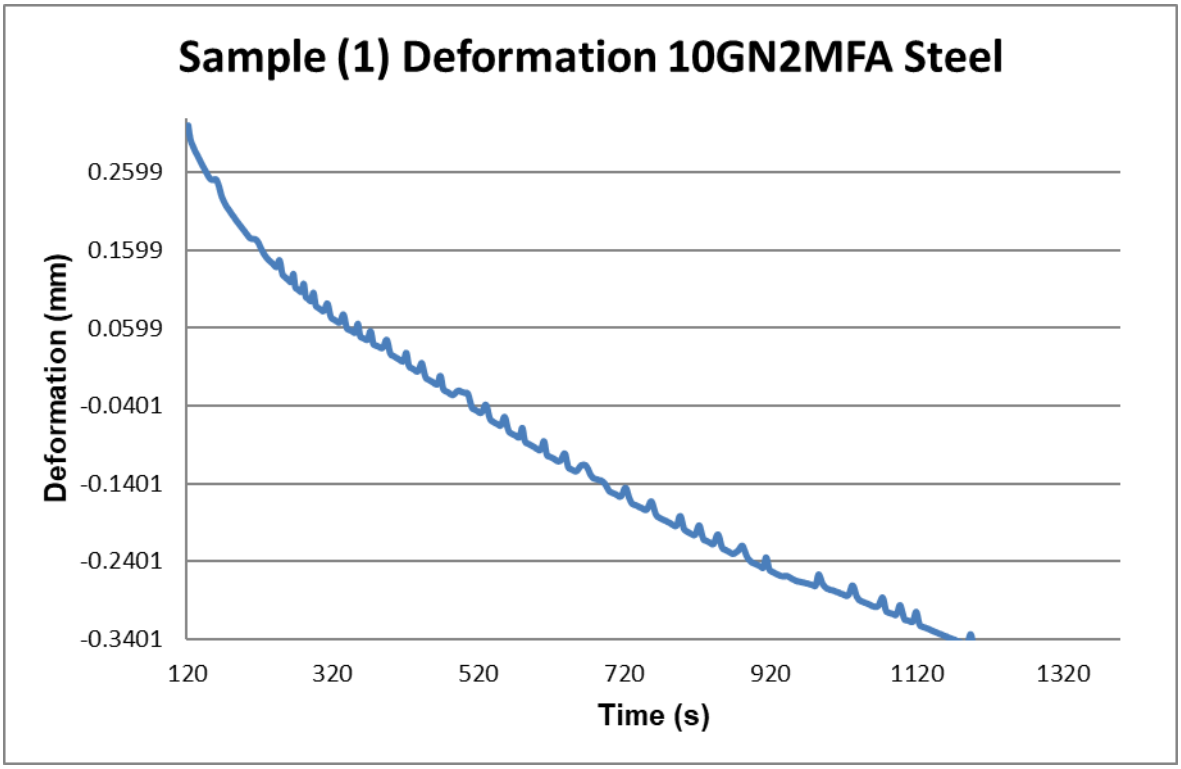
7. Reference

1. Robert W.. Principal of welding processes, physics, chemistry and metallurgy, ISBN –9780471253761. http://allaboutmetallurgy.com/wp/wpcontent/uploads/2017/02/Principles-of-Welding.pdf.
2. Eastering K.E. Introduction to the physical metallurgy of welding. ISBN- 978-0-7506-0394- https://books.google.cz/books?hl=en&lr=&id=DfwkBQAAQBAJ&oi=fnd&pg=PP1&dq=Introduction+to+physical+metallurgy+of+welding&ots=fXAYK6gn33&sig=xUgLqQ7UxW29yg1eUiHzURbyrEQ&redir_esc=y#v=onepage&q=Introduction%20to%20physical%20metallurgy%20of%20welding&f=false.
3. Moravec J., Dikovits M., Beal C., Nova I., Chandezon R., Sobotka J.. Selection of the proper diffusion welding parameter for the heterogenous joint Ti grade 2/AISI 316L. Manufacturing Technology, Volume 17, the Year 2017, ISSN 1213-2489.
4. J.J technical solution, Metal joining process: welding, riverting, bolting, brazing, soldering. Presentation, March 4, 2017
5. Kazakov N. F., Diffusion Boanding of Materials, 1985, Page- 304, ISBN: 9781483150550.
6. Rusnaldy. Diffusion bonding: An advance of material process. https://ejournal.undip.ac.id/index.php/rotasi/article/viewFile/2487/2199.
7. Lecture 17, nptl. Defination and description of different diffusion terms. http://nptel.ac.in/courses/113108052/module3/lecture17.pdf.
8. Somekawa H. and Higashi K., The Optimal Surface Roughness Condition on Diffusion Bonding, Materials Transactions, Vol. 44, No. 8 (2003), pp. 1640 to 1643. Online ISSN: 1347-5320.
9. Diffusion Bonding Editors: Stephenson, Springer Netherlands,1991, ISBN: 978-1-85166-591-4.
10. Fujiu Ke S. C., Zhou M., Bai Y., Atomistic investigation of the effects of temperature and surface roughness on diffusion bonding between Cu and Al, Acta Materialia, Vol 55, Issue 9, May 2007, pp 3169-3175. ISSN- 1359-6454.
11. Bhanumurthy K., Joyson D. and Jawale S. B., Diffusion Bonding of Nuclear Materials, BARC Newsletter; (no.331), Vol. 45, Issue 8, pp. 19-25. ISSN- 0976-21088.
12. Nguyentat T. Diffusion Bonding – An advance material process for aerospace technologies. Material science forum, 2000. https://www.vacets.org/vtic97/ttnguyen.htm.

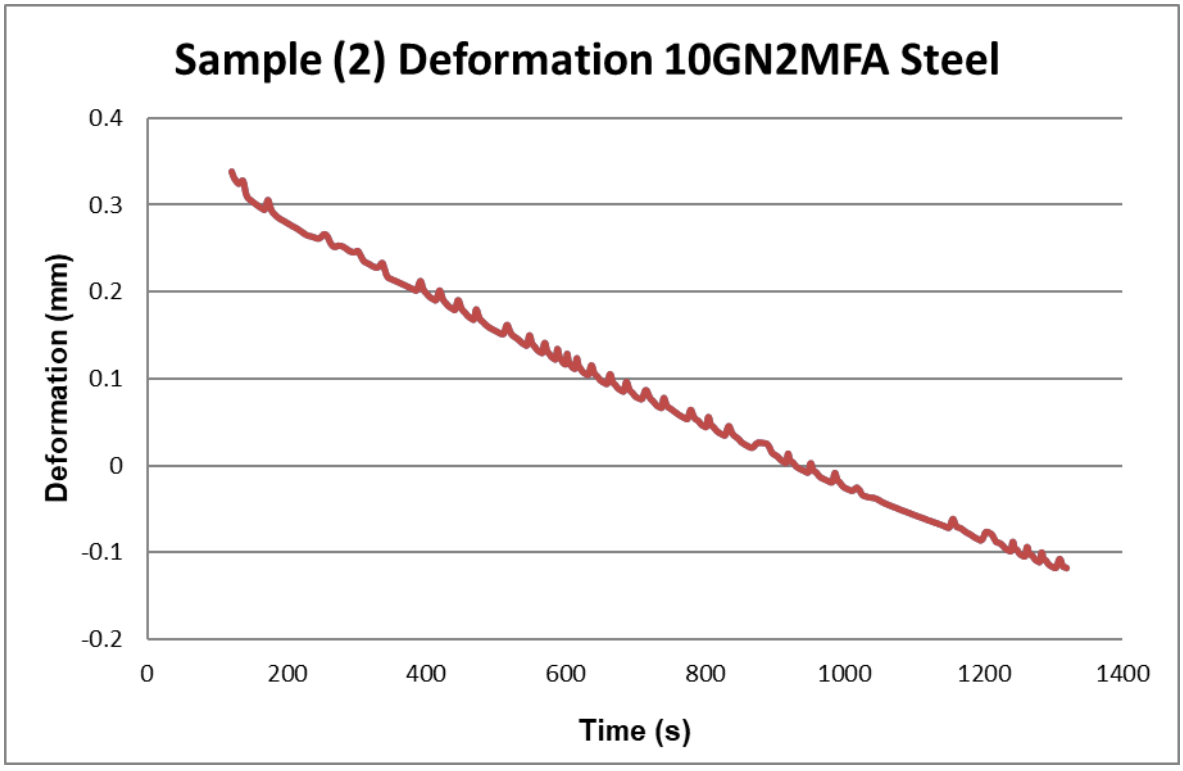
13. Rieley F.L.. Progress in nitrogen ceramics. Spring Science and bussines media, Pages., December 6, 2012. Pages 257-267. ISBN 9789400968516.
14. Akselsen. O.M., Review diffusion bonding of ceramics. The foundation for scientific and industrial research. Norway,24 April 1992, Volume 27, Issue 3, pp 569–579. ISSN -1573-4803.
15. Stephen D. The application of superplastic forming and diffusion bonding to air frame – Design and manufacture. International symposium on aeronautical science and technology of Indonesia. Jakarta, 1986.
16. Tinga, T. Principles of Loads and Failure Mechanisms. London: Springer Verlag. DOI: 10.1007/978-1-4471-4917-0, ISBN - 978-1-4471-4917-0.
17. Fong J. T., Fatigue mechanism. Springer, DOI: 10.1520/STP35912S, ISBN-EB - 978-0-8031-4744-7.
18. Dieter G. E.., Mechanical metallurgy. MCGraw-Hill, ISBN 0-07-100406-8.
19. Kailas S.V. Mechanical properties of metals. Class lecture for course Material Science. NPTEL. Indian Institute of Science, Bangalore, India. Retrieved from http://nptel.ac.in/courses/112108150/4.
20. A. Saxena, Nonlinear fracture mechanics for engineering, CRC press, LLC, Boca Raton, 1998. ISBN - 978-0849394966.
21. S. Anandh , Balani K. Fatigue presntation. IIT, kanapur.
22. CSN EN 3987, CESKA TECHNICKA NORMA. 31,2006
23. Mech4study,http://www.mech4study.com/2017/04/diffusion-bonding-principle-working-application-advantages-and-disadvantages.html. April 8, 2017
24. Matocha K., Čížek P., Kander L. and Pustějovský P., Resistance of 10GN2MFA-A Low Alloy Steel to Stress Corrosion Cracking in High Temperature Water., Published: September 26, 2011, ISBN 978-953-307-599-0.
25. Timofeev B. T. And Vasil'eva N. A., Influence Of Technological And Operational Factors On Fatigue Resistance Of 10gn2mfa Steel. Springer US, September 2014, Volume 46, Issue 5, pp 644–648, ISSN - 1573-9325.
26. Giginyak F. F. And Maslo O. M., A Relationship between Damage In 10gn2mfa Steel And Low-Cycle Strain-Controlled Loading At Different Deformation Frequencies. The strength of the material, 03 July 2017, Vol. 49, Issue 2, pp. 158 – 164, ISSN- 4902–034.
27. Makhnenko V.I., Markashova L.I., Makhnenko O., Berdnikova, Olena Shekera, V.M., Zubchenko A.S. The growth of Corrosion Cracks in Structural Steel 10GN2MFA. International association welding, pp. 2-5, 2012, Vol.8, ISSN- 301747120.
28. Gleeble users traning 2012, spring 2012, Dynamic systems Inc.

29. Navarro-López A., Hidalgo J., Sietsma J., Santofimia M.J., Navarro-López A., Hidalgo J., Sietsma J., Santofimia M.J., Characterization of banitic/martensite structures formed in isothermal treatments below the Ms temperature, Material charcterzation, Material charcterzation, Vol.128, Issue 9, June 2017, Pages 248-256. ISSN- 1044-580.
30. Manna R., Continuous Cooling Transformation (CCT) Diagrams, University of Cambridge.Presntation. https://www.phasetrans.msm.cam.ac.uk/2012/Manna/Part3.pdf.
31. Weber A., Klarner J., Vogl T., Schöngrundner R., Sam G. and Buchmayr B., Mechanical Properties of low alloy steels with banitic microstructures and Varying carbon content, Material Science Engineering, 2005. IOP Conference Series: Materials Science and Engineering 012005, Volume 119, 2016, conference 1. DOI - 10.1088/1757-899X/119/1/012005.
32. Podder A. S., Lonardelli I., Molinari A. and Bhadeshia H. K. D. H., Thermal Stability of Retained Austeinite in Banitic Steel: an in situ studies, The royal society, 2011, ISSN- 1471-2946, DOI - 10.1098/repa.2011.0212.
33. Fonda R.W, Vandermeer R.A, and Spands G., Continuous Cooling Transformation (CCT) Diagrams for Advance Navy Welding Consumables. Naval Research Labrotary, Report Number-NRL/MR/6324--98-8185, Page 31, July 15, 1998.
34. Vrba J., Moznosti Aplikace Difuzniho Svarovani U Materialu 10GN2MFA, Bachelors thesis, 2016.

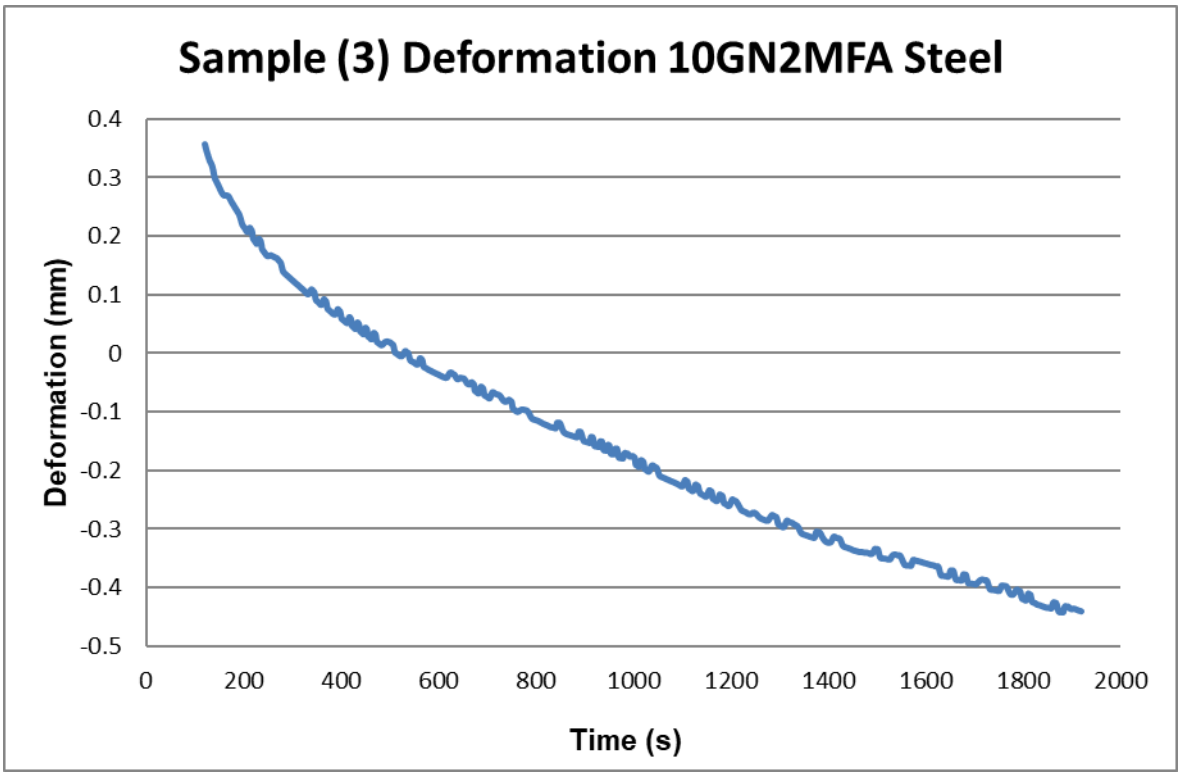
List of attachment



Attachment 1- Deformation the welding sample 1 at holding temp.



Attachment 2- Deformation the welding sample 2 at holding temp



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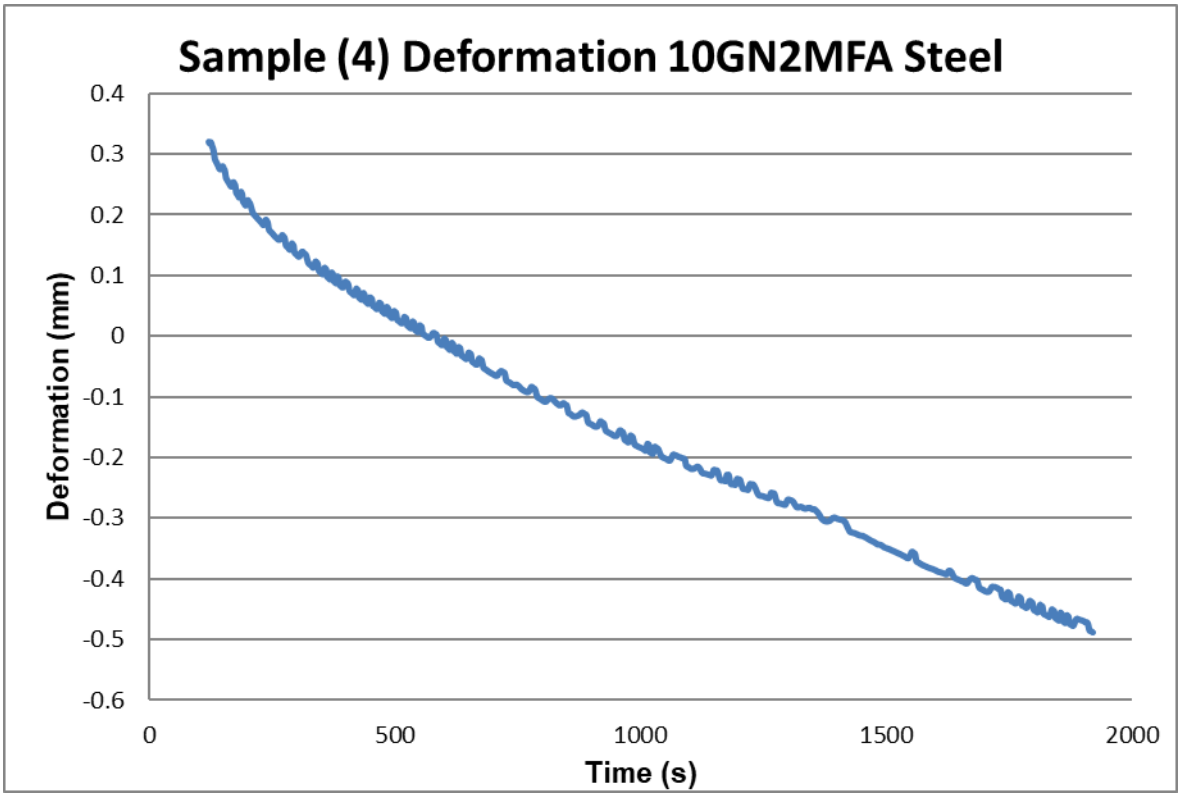
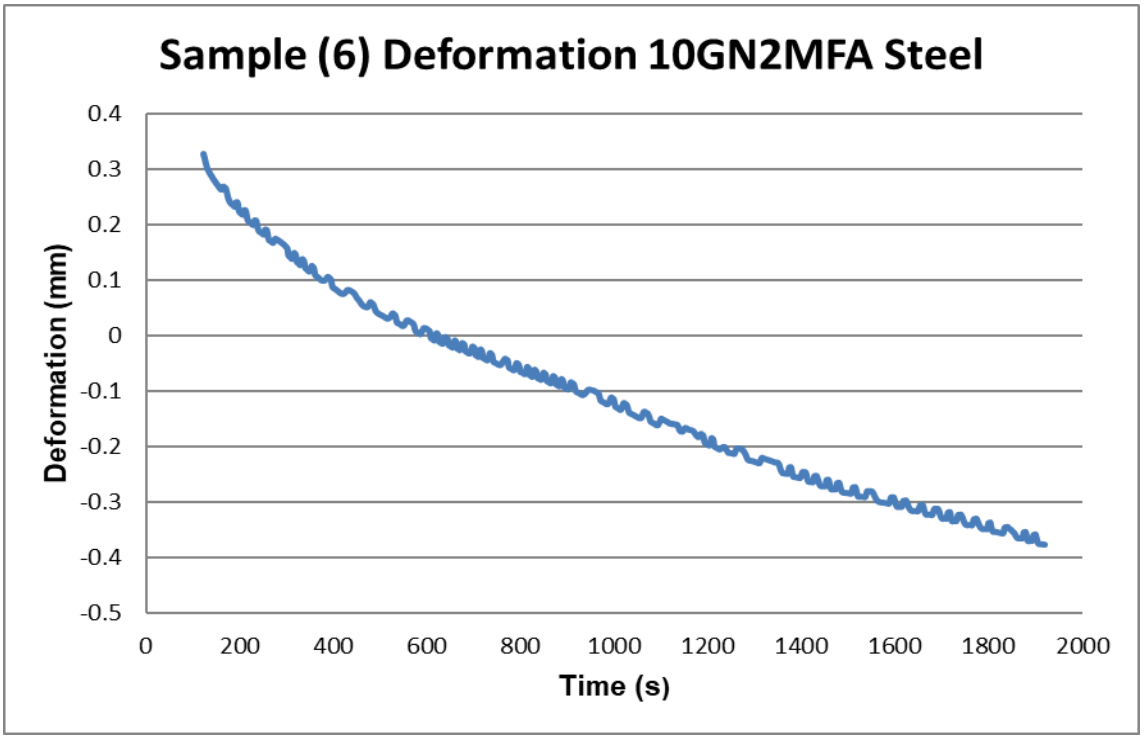
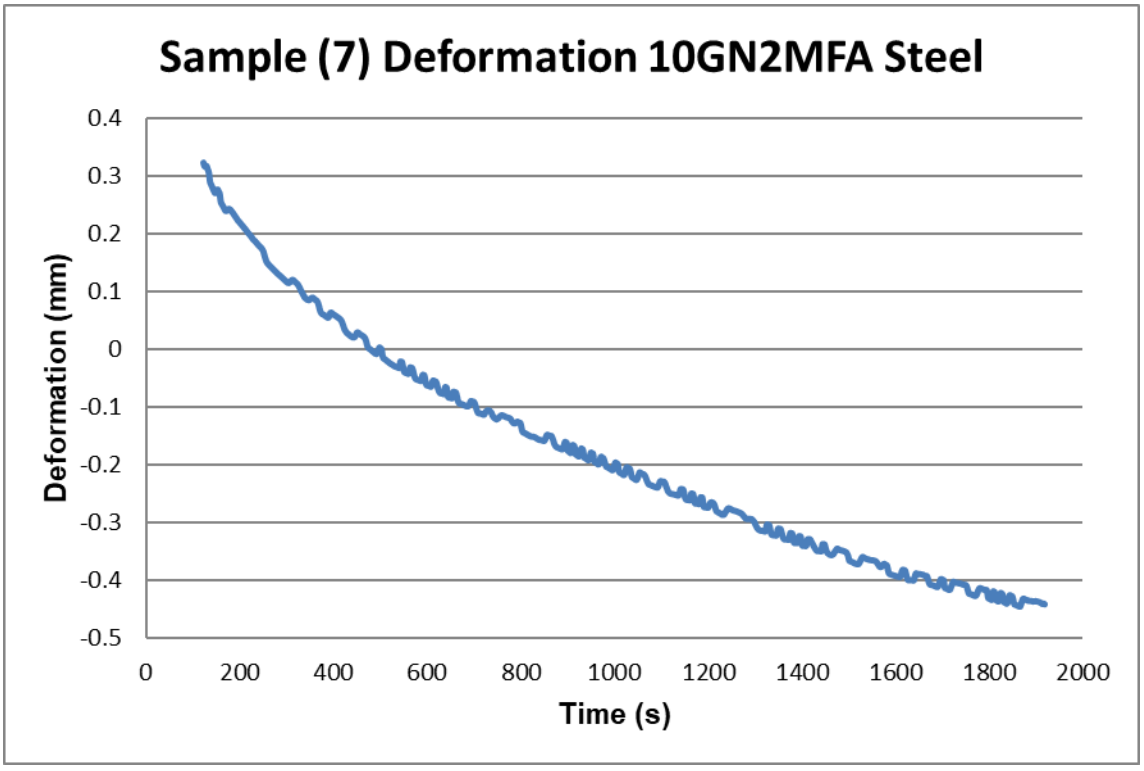


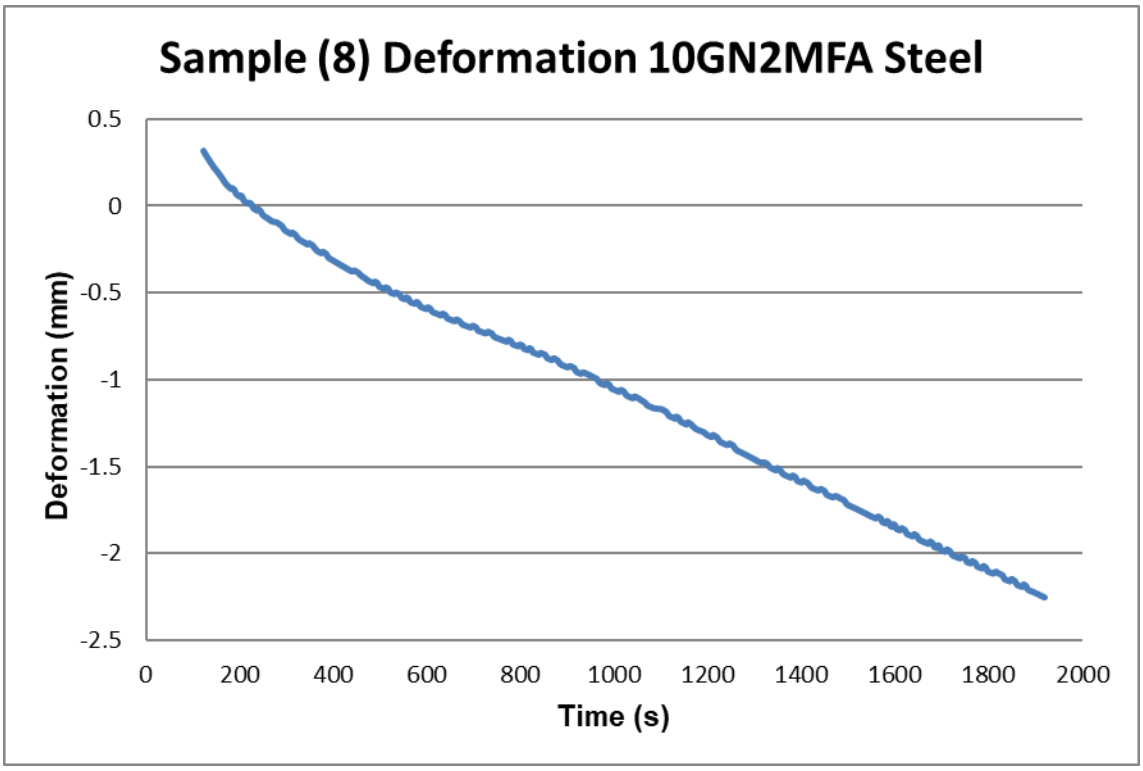
Attachment 4- Deformation the welding sample 4 at holding temp.



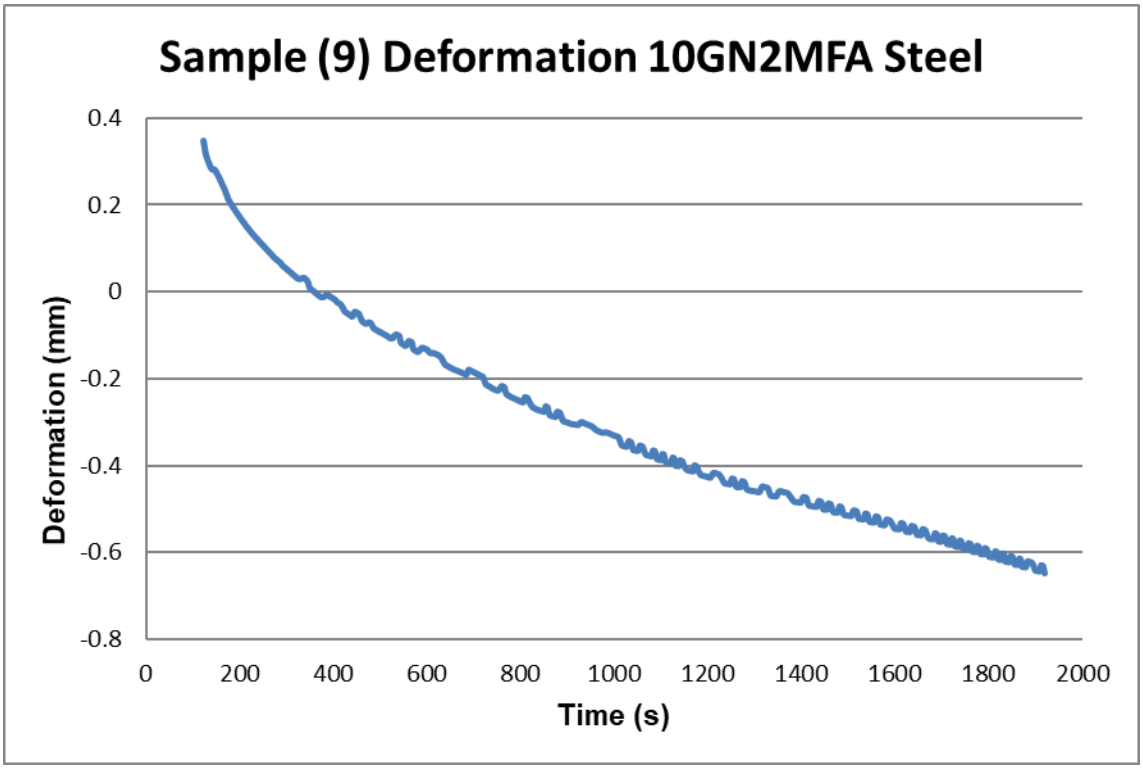
Attachment 5- Deformation the welding sample 6 at holding temp.



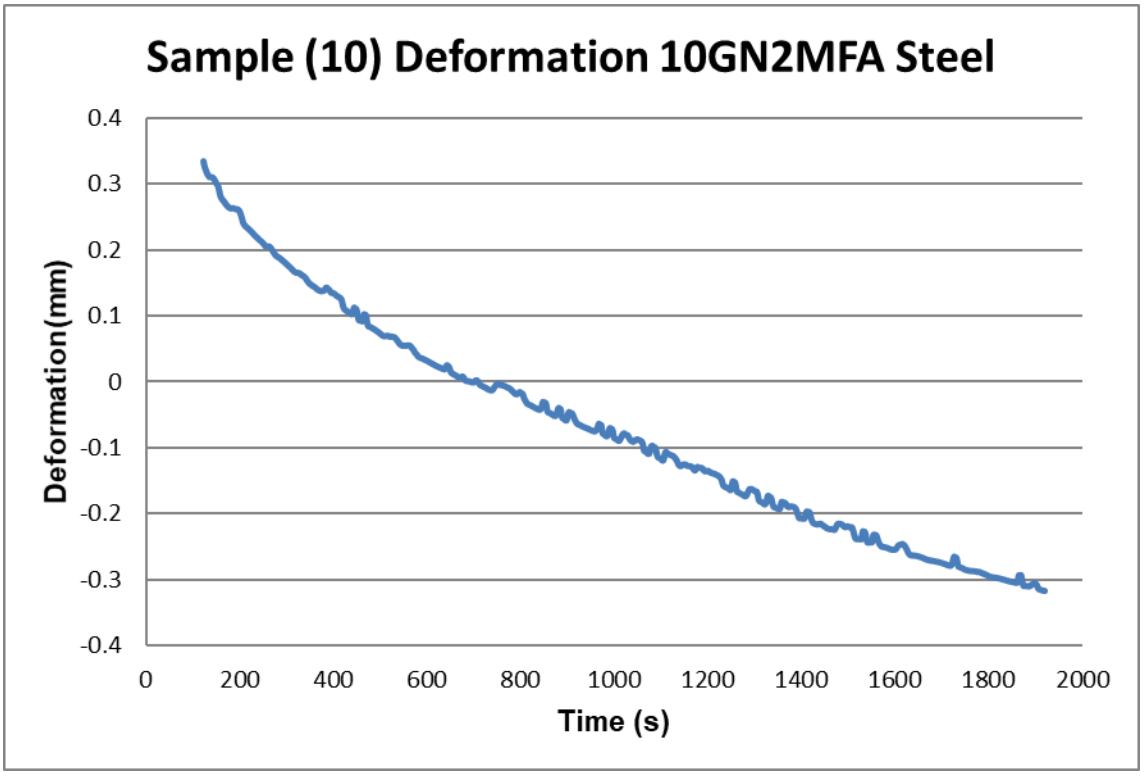
Attachment 6- Deformation the welding sample 7 at holding temp.



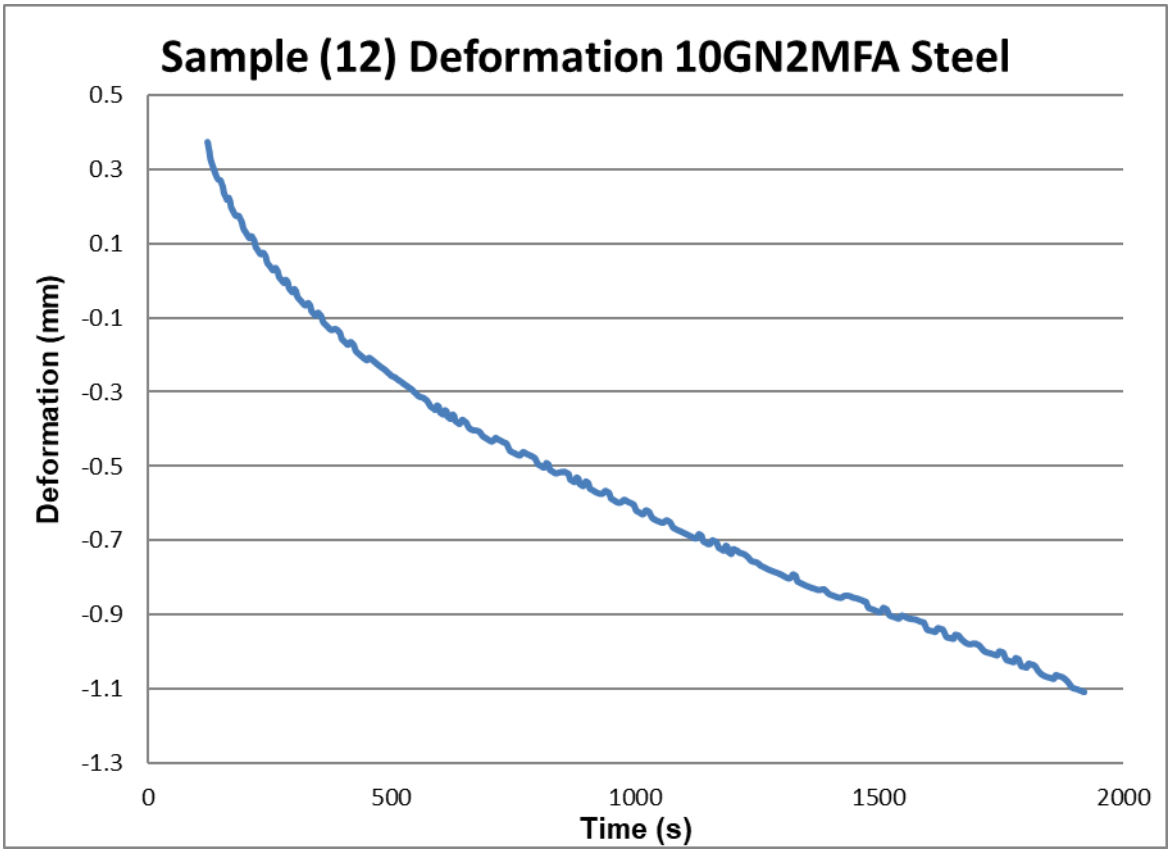
Attachment 7- Deformation the welding sample 8 at holding temp.



Attachment 8- Deformation the welding sample 9 at holding temp.



Attachment 9- Deformation the welding sample 10 at holding temp.



Attachment 10- Deformation the welding sample 12 at holding temp