

# Investigation of diffusion process activation in pinched surfaces

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A diffusion contact process was analyzed during welding without melting occurring in the active protective environment. The three basic activation mechanisms were determined as: impulse melting, chemical melting and contact melting. It was proven that it is possible to activate diffusion processes due to the high rate of reactions that are micro-explosions within micro-volumes on top of the pinched contacting surfaces. A well defined interval of heating values was found. This interval provides suitable conditions for diffusion contact of welded surfaces. Experimentally, it was found that the heating rate interval for diffusion welding of carbon steel and high-alloy steel is 130-200 °C/s.

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## 1. Introduction

Diffusion welding enables producing stress-relieved homogeneous connections of various materials. Usually, a diffusion contact of various metals may be achieved by pressure welding methods. In this process, two metals may be welded at a temperature lower than the melting points needed to join metals without adding some soldering material. The main parameter of such a process is the pressure that provides the plastic deformation to the extent needed to permanently join these metals.

A routine method of pressure welding is by using vacuum pressure [1]. However, sometimes this process may be achieved at an atmospheric pressure in a special protecting environment [2-8]. Pressure welding in vacuum has been extensively researched and described in detail [1,9-12]. However, the current literature lacks well-grounded data about the activation mechanisms of pressure welding in atmospheric pressure.

A major feature of pressure welding under atmospheric conditions is the application of a protective activation environment, called: Surface Activation Substance (SAS). Such surface activation substances in liquid or paste-like form are used to coat joined surfaces. SASs protect these surfaces from oxidation. Moreover, SASs help in reducing oxides and clean the joined surfaces. Various chemical reactions of SAS components and surface materials occur while heating. SASs are transformed to gaseous form at the welding temperature interval and are self-removed from the reaction zone. Chemical cleansing of metal surfaces activates and accelerates the diffusion contact [4,5,7-9]. Various organic compounds such as ethanol, glycerin, epoxies, and others may be applied by means of SAS.

In spite of the apparent simplicity of the pressure welding in an SAS environment, its application is difficult

due to insufficient investigation of activation processes. A main goal of this work is to investigate the activation processes involved in diffusion welding within SAS environment.

## 2. Surface phenomena analysis

### 2.1 Kinetic description of contacting materials

It is well known that a conventional kinetic sequence regarding the physical and chemical processes takes place during diffusion contact of various materials [1,10,11]. This process consists of three basic stages:

1. Physical interaction;
2. Chemical interaction;
3. Volume interaction in the contact zone.

The physical contact of two surfaces takes place in the first stage. This contact results in plastic deformation of micro-roughness under compressing forces. These forces prevail at the earliest stages of roughness deformation. Activation of contact surfaces and the formation of active centers occur in the second stage. This process begins under conditions of joint plastic deformation and a chemical reaction takes place. The welding temperature is the main factor governing the contact intensity of the developed attachments. At the third stage, strong chemical bonds are created. Diffusion processes and strength relaxation occur during this stage.

Diffusion processes in the contact area of different materials may result in melting of the contact [12,13]. This process has a commonly eutectic mechanism and may be associated with materials that are non-metallic. The volume interaction of contacting materials occurs in the third stage and results in the formation of common clusters in the contact zone and the relaxation of internal stresses. A volume diffusion interaction of different materials leads

to the formation of a transition zone. Such a zone consists of limited and unlimited solid solutions, eutectoids and inter-metals, as well as complex chemical compounds [10].

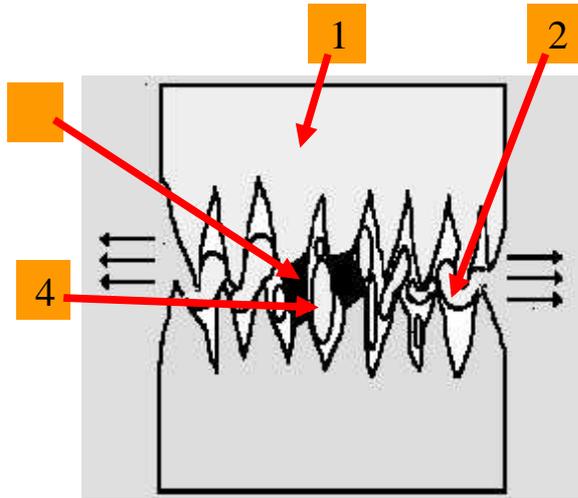


Fig. 1. Contacting surfaces. 1) Surface layer of a welded metal; 2) Gaseous pyrolysis products; 3) Heating zone of contacting surfaces; 4) Gas-bubbles filled with pyrolysis products.

## 2.2 Characteristics of hydrocarbonaceous binding agent (SAS) behavior

Hydrocarbon compounds inserted upon the contact surfaces contribute additional features to the diffusion activation processes:

1. The heating of hydrocarbon binding agents leads to their evaporation and pyrolysis. Active gaseous substances such as hydrogen, carbon, carbon oxide, and various carbon compounds are formed during these processes and reduce metal oxides. This significantly facilitates the diffusion processes.

2. Surfaces begin their contact at micro-ledges; therefore, a contacting surface area in the initial stage is only about 0.01-1% of all surface areas [14]. Fig. 1 illustrates contacting surfaces similar to the structure in ref [9]. Because of a high density of a current flow through the contacting micro-edges, this current is enough to rapidly heat and simultaneously cause deformation under an applied pressure. This deformation results in diminishing of roughness and increasing of the surface contact.

The heating rate of the micro-ledges may be estimated by approximating the surface roughness by means of triangular prisms of the type shown in Fig. 2:

- Roughness features (micro-ledges) are shown by equilateral triangles of height  $h$  (see Fig. 2a) that obey the normal distribution law with the following parameters:  $\bar{h}$  is an average of the distribution function and  $s$  is a standard deviation.

- The triangle is a side plane projection of a triangle prism (Fig. 2b) with a length of  $\delta = h$ .

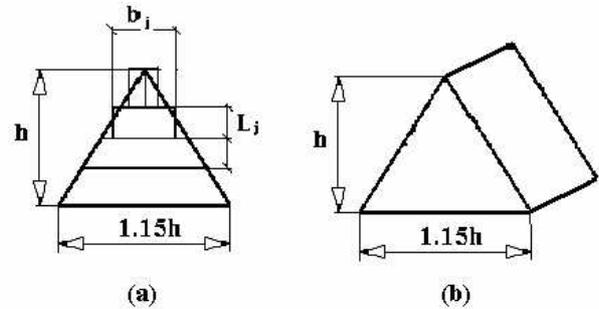


Fig. 2. Micro element profile approximation: (a) front view, (b) side view.

All calculations were done numerically using conjugated elements of a rectangular section as shown in Fig. 2. The height  $h$  of the triangle was divided to  $n$  short segments. Therefore, the dimensions of the conjugated elements are as follows:

$L_j = h/n$ ,  $\delta$ , and  $b_j = 1.15L(2j - 1) / 2$ , where  $j$  is the current number of the conjugated elements and  $1 \leq j \leq n$ .

An example of resistive heating is described by the following equation:

$$Q_j = I^2 \rho \frac{L_j}{b_j \delta} \tau, \quad (1)$$

where  $Q$  is the energy,  $I$  is the current going through the materials,  $\rho$  is the resistivity, and  $\tau$  is the heating time.

The heating temperature may be calculated using a standard formula:

$$t = \frac{Q_j}{cm_j}, \quad (2)$$

where  $c$  is a heat capacity,  $m_j$  is the  $j^{\text{th}}$  element mass ( $m_j = L_j \delta b_j \gamma$ ), and  $\gamma$  is the density of the welded material.

A current value was chosen based the following assumptions:

- Typical current density is 8 – 15 A/mm<sup>2</sup> in such processes,
- All dimensions are Geometrical,
- Statistical parameters of roughness were chosen with  $\bar{h} = 25 \mu\text{m}$  and  $s = 5 \mu\text{m}$ ,
- Initial contact area was 1% of all attached surfaces.

If we chose the roughness dimensions with a confident probability of 0.997, then the current value will be less than  $I = 0.1$  A.

We used the following material parameters:

$$\begin{aligned} c &= 0.465 \text{ kJ/kg}\cdot\text{K}, \\ \rho &= 10^{-7} \Omega\cdot\text{m}, \\ \gamma &= 7850 \text{ kg/m}^3. \end{aligned}$$

The major items of importance revealed in our analysis were as follows:

- The boiling temperature of the hydrocarbonaceous binding agent (SAS) is, for example, 78 °C for ethanol, or 290 °C for glycerin.
- The temperature of the phase transition of steel finishes at the level of 900 °C, in other words, a transition to a monotonous decreases the deformation resistance.
- A melting point of 1535 °C.
- A boiling temperature for iron of 2735 °C.

Various layers of various thicknesses were calculated, e.g.,  $5 \leq n \leq 250$ .

Our calculation showed that the material comprising the micro-ledges may overcome the conditions of boiling in the upper layer with a thickness of  $\sim 100$  nm during a short period of 100 ns (see Fig. 3a for one of the calculations of the variances). Such a heating rate relates to the explosion conditions in the micro-volume. This conclusion is in agreement with experimental and estimated data described by V. I. Oreshkin et al. [15,16]. Our calculated outcomes are in agreement qualitatively with the described results concerning the mass, energy density, and the time that the micro explosion occurred. Therefore, micro-explosions of micro-ledge material are possible while the material is being heated.

3. The estimated time of the micro-ledges heating up to the temperature of SAS boiling was shown to be about  $10^{-4}$  s (see Fig. 3b for example, which was done for glycerin). Therefore, the boiling conditions for SAS are favorable to create vapor micro-bubbles.

If the high rate of the heating and the prominent temperature gradients in the liquid phase are taken into account, then it is reasonable to assume that the creation of SAS micro-bubbles is followed by their collapse. Here, due to the high rate of metal being heated, the nearest layer of SAS to the metal surface gets enough energy for transfer to the superheated liquid state (for example, 243 °C and 6.38 MPa for ethanol).

4. Liquid, gaseous, or superheated substances situated between the pinched surfaces, form an environment with specific properties. This environment activates the material surfaces, which in turn accelerate diffusion processes. Active substances on the surface are in the form of liquid in the initial stage of the process. Therefore, micro-explosions of roughness on the upper area lead to blast waves within the liquid that influence the metal surface. The vapor bubbles that appear as the SAS is rapidly heated, explode in the liquid and also produce blast waves in the micro-volumes. During rapid heating, the liquid phase of the contact surfaces is rapidly transferred to a gaseous form or overheated liquid state. Thus after a short while, only micro-explosions of micro-ledges continue to produce the blast waves.

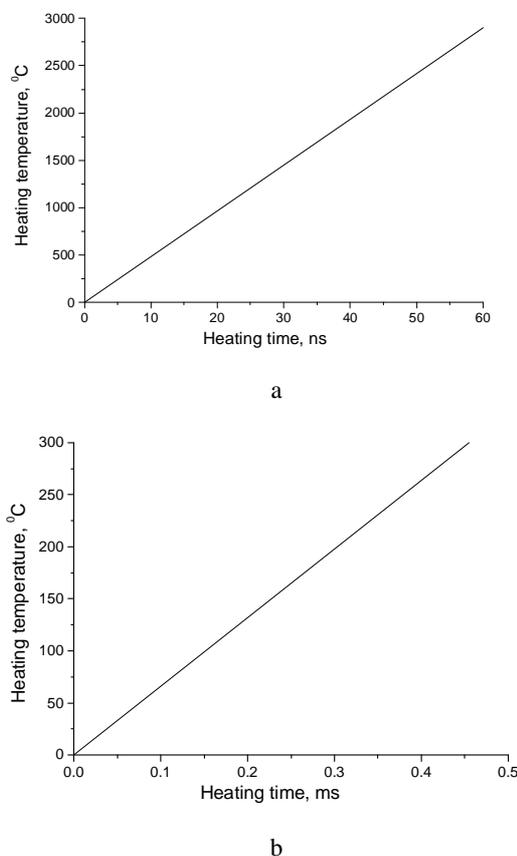


Fig. 3. Estimated heating time up to boiling temperature: (a) for iron, (b) for glycerin.

These blasts are a source of impulse stresses within the surface layers of the welded materials. Such impulse loads constitute a significant factor that influences the diffusion acceleration [18-24]. Measurements were provided under various conditions: explosion, ultra-sound vibration, electro-pulse treatment, and plasma treatment. This blast influence was explained as follows: surface dislocations capture arbitrary diffusing atoms and then move them to the surface. Therefore, this phenomena accelerate the diffusion process.

5. SAS pyrolysis on metal surfaces results in the formation of active substances such as hydrogen, carbon, carbon oxide, and other carbon-based compounds. These active substances promote the appearance of the contact melting effect. The incipient liquid phase wets the surfaces in contact and leads to a decrease in the surface tension within the welded materials. Therefore, activating the surface accelerates the diffusion process.

### 2.3 Influence of an active-adsorption environment on diffusion processes

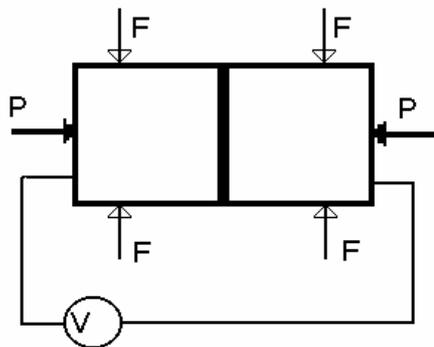
Plastic deformation of crystal solids is strongly related to dislocations appearance and translation. SAS does not create novel defects in the solid; however, it governs their appearance and growth. Products of SAS thermal

destruction are known to enable the plastification of crystal surfaces. These products accumulate in the contact zone and facilitate the migration and multiplication of dislocations on top of metallic friction surfaces. This effect was discovered by P.A. Rebinder [26]. Adsorption-active environments always decrease the surface energy and enable the development of new surfaces. If a same constant strain is applied to the surface, then the plastic deformation rate increases. As a result, the yield point of the material decreases and a resistance to deformation decreases, also due to a constant deformation rate [25]. Moreover, SAS influences the formation of dislocations in metals and in diffusion processes [27]. The various atoms from the diffusion create local displacements in the crystal lattice. These displacements assemble within the diffusion flow zones and create fields of elastic stresses. This results in the formation of dislocations or redistribution that results in the relaxation of the field of the elastic stresses.

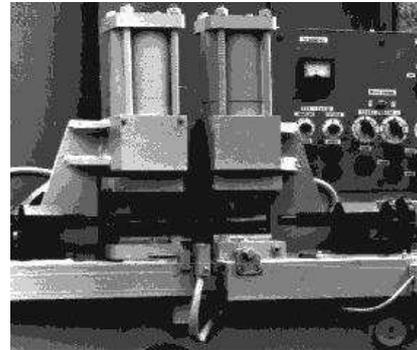
The dislocations create so-called “short circulation ways” that are zones of irregular diffusion. Here, the resistance to the diffusion decreases and a total flow of diffusant increases. The formation of dislocations and their redistribution in the diffusion layer accelerates the penetration of the diffusant into the target material.

Surface tension decreases because of the adsorption of SAS. An energy evolves owing to the decrease in surface tension, which enables the Rebinder effect. This energy creates the formation of novel dislocations in the diffusion layer. The Rebinder effect may be accompanied by enhanced dislocation density in the surface zone of metals, alloys, and other solid materials.

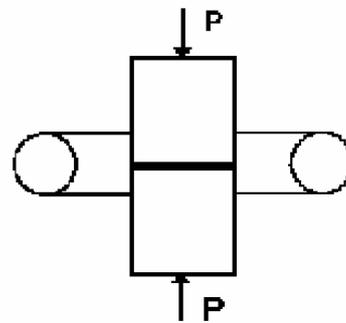
An analysis of the described above may be concluded in the following way: A pressure welding in a SAS environment may be characterized by additional specific activation mechanisms such as chemical and impulse processes. The chemical mechanism consists of gaseous products of SAS pyrolysis and is influenced by overheated liquid. The impulse mechanism is involved in the impact of blast waves resulting from micro-explosions. Moreover, the contact melting effect is also influenced by the diffusion processes. Thus, the experimental research objective is in analyzing these specific activation mechanisms.



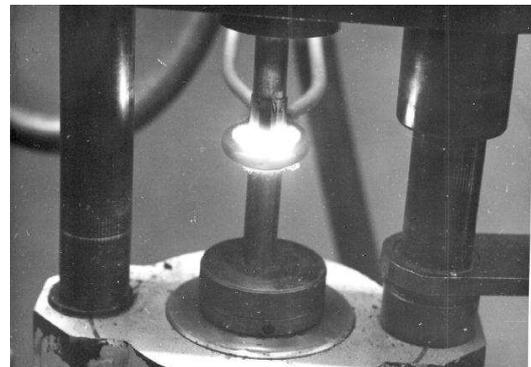
(a)



(b)



(c)



(d)

Fig. 4. Diffusion welding apparatus: a. Principle setup of a resistive heater; b. Side view of a resistive heater; c. Principle setup of inductive heater; d. Side view of inductive heater.

### 3. Experimental details

All experimental investigations were done using special laboratory machines, presented in Fig. 4. Fig. 4a shows a setup of the process of resistive heating. A side view of this setup is shown in Fig. 4b. Fig. 4c presents a setup for the contact formed by the induced heating of the joined metals. A side view of this system is shown in Fig. 4d. Various types of steels were used in our experiments:

carbon steel ( $C = 0.45\%$ ) and high-alloy steel ( $Cr = 4\%$ ,  $W = 6.5\%$ ,  $V = 2.1\%$ ,  $Co = 4.9\%$ , and  $Mo = 5.1\%$ ). The experimental samples were cylindrical and tubular forms with a round or tubular cross-section. Part of the tubular samples had the closed second ends as shown in Fig. 5a. These samples were used for analyzing the resulted pyrolysis products during the welding process. We studied the condensate formed on the internal surfaces of these closed samples, as shown in Fig. 5b. The experimental samples were 15 mm in diameter. Each welding experiment was repeated four times.

The liquid or paste-like material consisted of glycerin, ethanol, epoxy, or refined coal oil that was applied to create the surface active substance (SAS). SAS was coated on the open sides of the sample and then the diffusion welding was applied. Following that, the resulting hollow sample was slit open and the condensate material produced during the welding and following the cooling of the internal surfaces of the samples was studied.

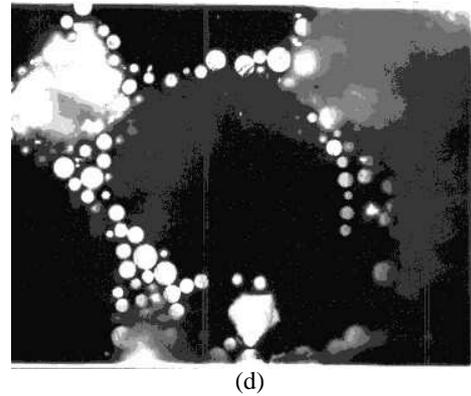
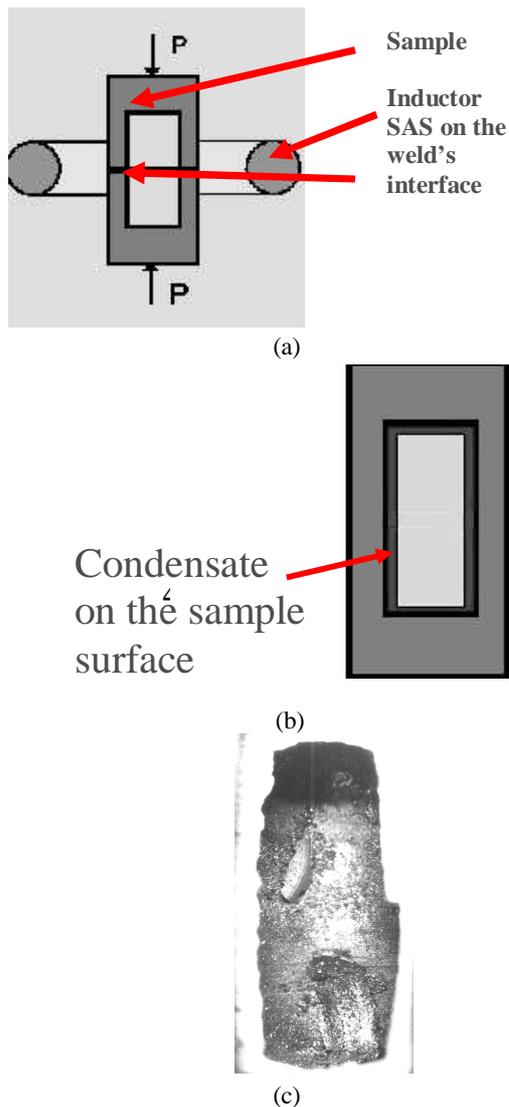


Fig. 5. Contact melting verification experiment. a) Principle heating setup; b) Pyrolysis condensate products; c) Fragment of the condensate removed from the sample internal surface; d) Microstructure of the condensate, containing spherical impurities.

Heating rate was controlled during the experimental trials from  $100\text{ }^{\circ}\text{C}/\text{sec}$  up to  $250\text{ }^{\circ}\text{C}/\text{sec}$ . The maximal heating temperature was no more than  $1200\text{ }^{\circ}\text{C}/\text{sec}$ , which is much lower than the melting point. The obtained welded connections were mechanically tested by bending and stretching. Destruction of the sample on the base material but not on the diffusion connection was used as a criterion in assessing the quality. The following testing machines were utilized for our tests: PM-50 (with the strain up to 500 kN) and H100 K-S (up to 100 kN). The structure of the micro- and macro-parameters were investigated using the following microscopes: optical microscope NEOPHOT-21, scanning electron microscopes JEOL JSM-840, and REMMA-202 microscope.

## 4. Experimental results and discussion

### 4.1. Melting of contacts

The material removed from the attached samples was studied. This material constituted condensate from the pyrolysis products. The condensate was shaped similarly to the internal form of the samples, as shown in Fig. 5c. This confirms that the material was enough viscose during the welding process.

Structure investigation of the condensate showed the appearance of spherical metallic impurities (see Fig. 5d). Only one source of these impurities exists inside the attached samples: this is the metal of the samples. The temperature of the samples was no more than  $1200\text{ }^{\circ}\text{C}$  and the metals were not included in the SAS material. Therefore, it can be seen that the contact melting mechanism is operational at the time of diffusion welding within the active protective environment. The presence of a liquid phase in the micro-volumes enhances the surface activation and diffusion acceleration processes.

#### 4.2 Influence of the SAS impulse

The same samples consisting of two different steels were welded by two different methods: resistive heating and induction heating. Measured time of the welding was 12 sec for resistive heating and 20 sec for induction heating with the same quality of the welded joints. Fig. 6 presents microstructures of these joints. It can be seen that these joints are of diffusion types:

- The crystallization structures are absent in the photographs, that is, the metal of the welding samples has not melt.

- There is an exact transition line.
- There is a ferrite layer on the border of the high-alloy steel. Such layers in the welded joints are typical of pressure welding in the vacuum of ferrite-pearlite steels [1,17].

The welding time difference in our experiments for various heating methods can be explained by considering the various heat sources and heat transfer features:

##### 1. Induction heating

- This is not a micro-explosion process of the surface roughness. Heating takes place uniformly for all contact surfaces. A heat transfer to the SAS also occurs at all contact surfaces.

- A micro-explosion of gas bubbles is the activation mechanism at the initial stage of the process (this mechanism acts until the SAS liquid phase starts).

##### 2. Resistive heating.

- A resistive heating occurs on the contact areas that appear only at 1% of all the contact surfaces at the initial stage and increases to 100 % during the welding process. Fig. 7 illustrates this phenomenon. The sample that appears here represents the case of a broken welding process.

- An impulse activation mechanism that initiates the micro-explosion of roughness on top and a micro-explosion of gas bubbles at the initial stage, is activated here.

Therefore, two different impulse mechanisms act in the case of resistive heating but only one mechanism forms the welded joint at the inductive welding. This difference results in increasing the duration of the process. Moreover, the significant influence of the various activation mechanisms validates the importance of impulse activation in general.

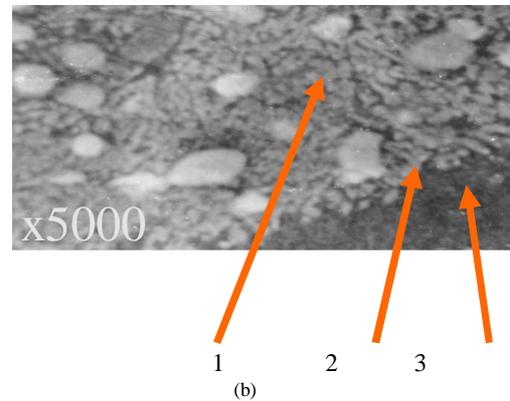
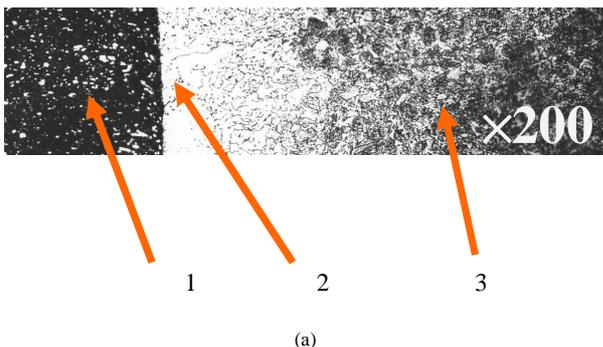


Fig. 6. Microstructure photography of the diffusion joined metals: (a) optical microscope, (b) scanning electron microscope. 1. High - alloy steel; 2. Boundary between two types of steel; 3. Carbon steel.

#### 4.3. Heating rate and reduction of oxides

The organic material (SAS) between two contact surfaces undergo pyrolysis, resulting in the formation of gaseous products such as hydrogen, carbon, and various carbon compounds, which are highly active during the heat state. These products adsorb on the solid metal oxides and react with the oxygen from oxides. This leads to the formation of gaseous substances removed during the welding process. Cleaned metal surfaces are observed this way and contact is made up to the end of the process. Several initial contacting zones are developed during the process, up to a full capture of the whole surfaces (Fig. 7).

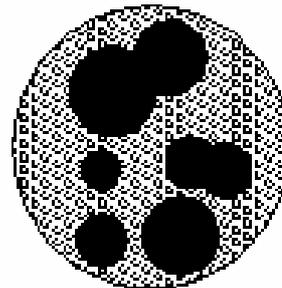


Fig. 7. Contact areas developed during diffusion welding (process was broken).

Heating temperature is an important factor of the welding process. The energy taken up in the process serves to operate the activation and diffusion mechanisms. In addition, the heating rate markedly influences the quality of the welded joint. Our experimental investigations have shown that a heating rate lower than 130 °C/sec is insufficient to provide a stable quality for the joints. The samples prepared with a low heating rate were destroyed on the contact line during bending tests. The heat diffusion from the zones of contact prevent the consumption of heat at these heating rates and diffusion of metals occurs non-

uniformly, which results in an insufficient contact strength. A heating rate of more than 200 °C/sec leads to the deterioration of contact and even to the destruction of samples, as shown in Fig. 8.

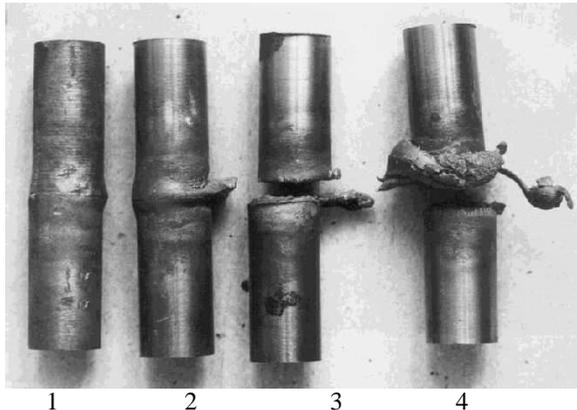


Fig. 8. Heating rate influence on quality of welding: (1) 200 °C/sec, (2) 210 °C/sec, (3) 220 °C/sec, (4) 250 °C/sec.

Destruction of samples, which appeared during the welding process, may be explained as follows: gases and liquids should be removed while heating.

All of the processes: pyrolysis, chemical reactions, diffusion joints formation, are simultaneous; therefore, it is possible to have closed micro-volumes in which the gas pressure may be significantly increased. This process may lead to the creation of over-heated liquids in these volumes. At the same time, the zones of contact with diffusion joints formed (see Fig. 7) are systems where mechanical tensions are redistributed at all times. Due to the high pressure and indefinite tensions, the redistributions of the closed volumes burst open and gaseous products evacuate from them, resulting in the formation of welded joints. Fig. 9 shows an example of such a micro-volume. A burst occurred late and the contact formation was consequently not finished.

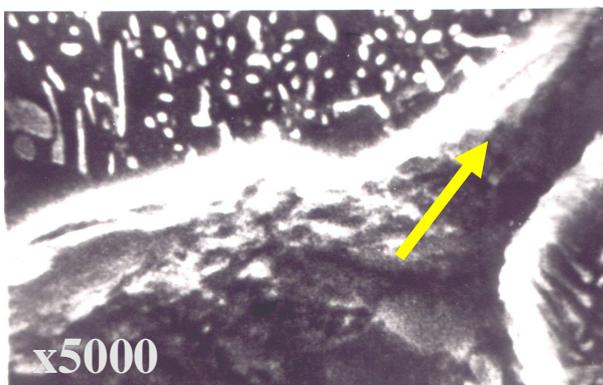


Fig. 9. A cavity filled by pyrolysis products, the arrow indicates on the pyrolysis products flow.

The high rate of formation of joints on separate sites raises the probability of formation of large closed volumes. A burst of these volumes occurs at the highest heating temperatures. This situation is possible at high heating rates (Fig. 8). Thus, a heating rate from 130 to 200 °C/sec provides stable conditions for the formation of quality diffusion joints.

Therefore, heating enables the SAS pyrolysis and/or transfer to the overheated liquid state. The components of SAS formed as a result of the heating adsorb on the metal oxide surfaces and reduce them, thus providing activation of cleaned surfaces. There is a well defined interval of heating rates that enhances stable and balanced conditions for activation of surfaces and formation of diffusion joints. The diffusion joint is not formed on all surface sections or is destroyed if the heating rate is not in this recommended interval.

## 5. Conclusions

In this work it was shown that the basic activation mechanisms of pressure welding in the active protective environment (SAS) are: impulse, chemical, and contact melting.

Also:

1. The activation of surfaces and the processes of diffusion accelerate due to micro-explosions on pinched surfaces, was demonstrated.
2. The contact melting of micro-parts from pinched surfaces was proven.
3. The existence of a well defined interval of heating values to provide stable and balanced conditions for diffusion contact of welded surfaces, was shown.
4. Experimentally, the heating values interval for diffusion welding of carbon steel and high-alloy steel was found to be 130-200 °C/s.

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