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CONSIDERATION ABOUT HIGH-PRESSURE REACTORS WITH CYLINDRICAL SYMMETRY

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Reactoarele de presiune înaltă utilizate în scopuri industriale sunt fabricate în construcție fretată. Cele mai apropiate componente de capsula de sinteză (matrița şi cele două dornuri) sunt fabricate din WC-Co sinterizată. Aceste componente sunt pretensionate cu inele din oțel. În această lucrare sunt prezentate rezultatele experimentale efectuate pentru a stabili corelații între proprietățile fizico-mecanice ale materialelor din care sunt fabricate matrițele şi deformațiile specifice elastice ale matriței din WC-Co în timpul procesului de fabricare a reactoarelor de presiune înaltă.

High-pressure reactors used in industrial purpose are manufactured in compound construction. The closest components to the synthesis capsule (a die and two anvils) are made from WC-Co alloy. These components are binded with steel rings. In this paper the experiments made to establish a correlation between the physical-mechanical properties of tungsten carbide dies and the linear relations are presented. The plastic deformation of the die made from tungsten carbide resulted in the assembly process is shown.

Keywords: High-pressure reactors, die, multilayer, WC-Co alloy.

1. Introduction

High-pressure reactors are used in research and industrial purposes in synthetic diamond production. The pressure is above 4,5 GPa and temperature is above 1450^{o}C. The most important parts of the reactors are the die and the anvils (fig. 1 and fig. 2). These are manufactured using WC-Co alloys and are pre-tensioned with a series of annular rings [1].

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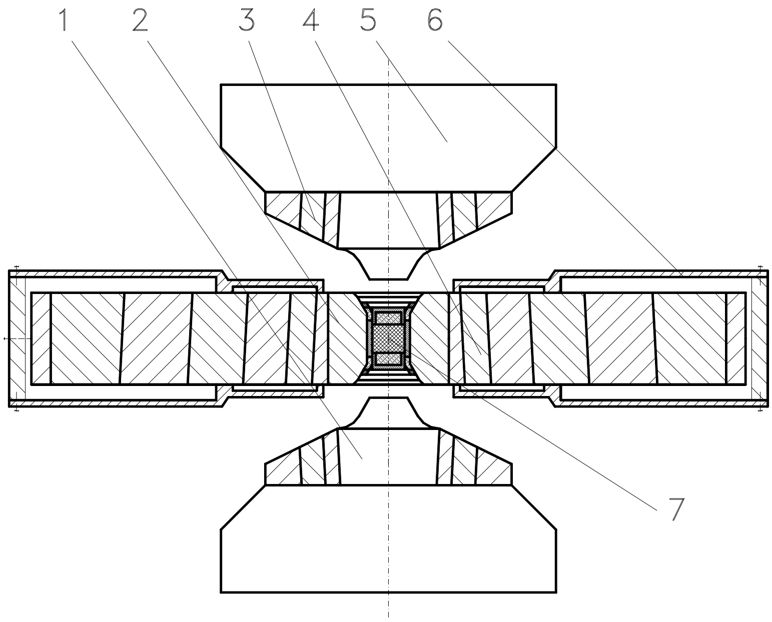

Fig. 1. Ultra high pressure and high temperature reactor; 1 - Bottom anvil with binding rings; 2 -WC-Co die; 3 - Top anvil with binding rings; 4 - Binding steel rings; 5 - Support anvil block; 6 - Cooling jacket; 7 - Synthesis capsule.

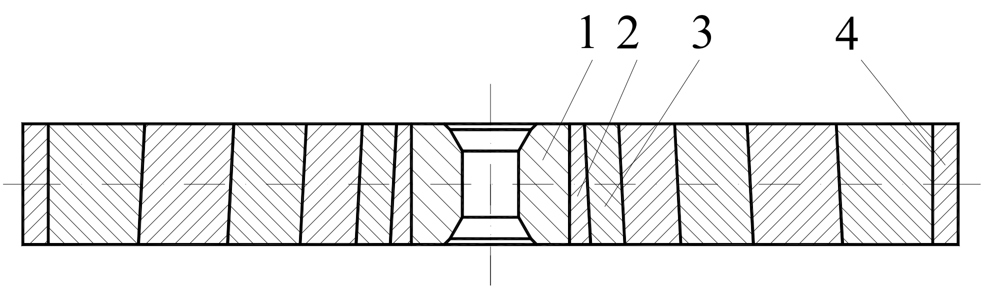

Fig. 2. Die prestressed with rings. 1 - Die; 2 - First ring (cylindrical at inner surface and conical at the exterior surface); 3 –Rings with conical surface, using pressing for assembling. 4 - Protection ring manufactured from soft steels.

In the construction of the die-binding rings assembly, the level of pressure on the external surface of the die is very important. If this pressure is low when the service pressure is present the number of the service cycles decrease. If this pressure is very high, the die may fail during the assembly process. The direct and simple measurement of the degree of pre-stressing of the die is made by measuring the difference between the inner diameter of the die before and after the assembling the binding rings.

The diagrams for general synthesis cycles used in diamond synthetic production [2-4] are represented in fig. 3. In this figure the pressure is the hydraulic pressure in the hydraulic cylinder of the press not the synthesis pressure.

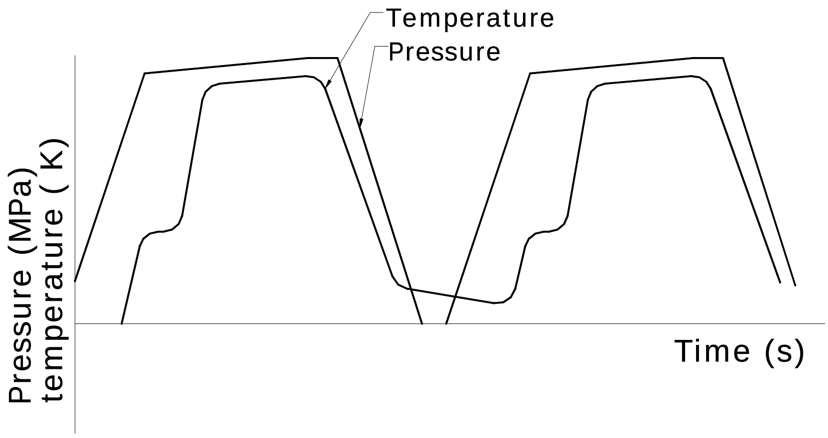

Fig. 3. Cyclic variation of pressure and temperature in the case of reactors for diamond synthesis [2-4]

Fig. 4 contains the relative number of loading cycles of the dies used in service conditions versus the actual strain of the inner diameter of the dies. The relative number of cycles of the dies is:

$$N_R = \frac{N_i}{N_{\max}} \cdot 100\% \qquad (1)$$

where: N_R is the relative number of cycles; N_i is the actual value of the number of the loading cycles of the dies used in service conditions; N_{max} is the maximum values of the number of the cycles of the dies used in service conditions.

From figure 4 two important conclusions result:

- the pressure value on the external surface of the die has a great influence on the life-time of the dies;
- there are different maximum relative numbers of the loading cycles of the dies used in service conditions for different grade of tungsten carbide of the dies.

In the paper the following specifications we use:“P-R” WC-Co powder from Plansee SE Company, dies in Dacia Synthetic Diamond Factory, Romania sintered; “B” dies from Board Longyear Company; “S” dies from Sandvik AB Company.

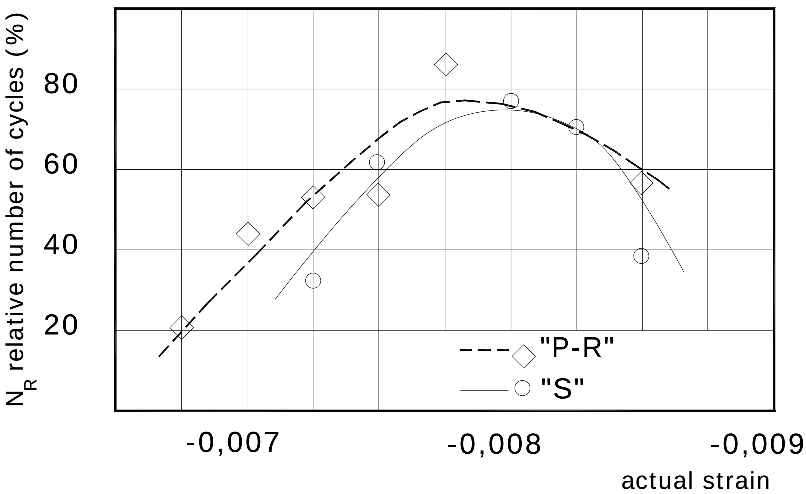

Fig. 4. The relative number of loading cycles of the dies used in service conditions versus the actual strain of the inner diameter of the dies [5,6].

2. Theoretical considerations

Consider a multilayer construction shown in fig. 5. The layers are made from different materials having different elastic properties. Generally the real construction is made from minimum 4 different materials. The die is made from WC-Co alloy, ring number 1 is made from hard steel, rings number 2, …, (n) are made from steel with high strength and ring number (n+1) – safety ring, is made from soft steel [5,6].

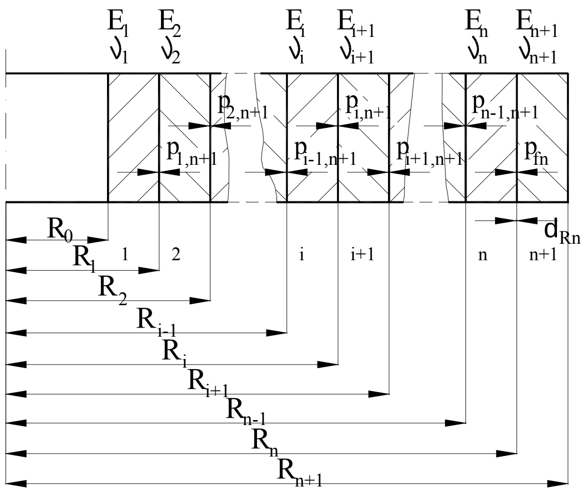

Fig. 5. Multilayer construction from (n+1) rings with different material properties.

The rings of the thick walled cylinders are considered. The Lamé equations are used and the displacement equations are writing.

We write the continuity equations for all rings contact surfaces. For the contact surfaces between the ring (i+1) and (i) this equations becomes [7]:

$$\frac{1-\nu_i}{E_i}\frac{R^2{}_{i-1}\cdot p_{i-1,n+1}-R^2{}_i\cdot p_{i,n+1}}{R^2{}_i-R^2{}_{i-1}}R_i+\frac{1+\nu_i}{E_i}\frac{R^2{}_{i-1}\cdot R_i\cdot(p_{i-1,n+1}-p_{i,n+1})}{R^2{}_i-R^2{}_{i-1}}=$$
$$=\frac{1-\nu_{i+1}}{E_{i+1}}\frac{R^2{}_i p_{i,n+1}-R^2{}_{i+1}p_{i+1,n+1}}{R^2{}_{i+1}-R^2{}_i}R_i+\frac{1+\nu_{i+1}}{E_{i+1}}\frac{R^2{}_i R_{i+1}(p_{i,n+1}-p_{i+1,n+1})}{R^2{}_{i+1}-R^2{}_i} \quad (2)$$

where i=1,2,3,..,(n-1), and n>2; E - Young modulus, ν - Poisson ratio, R_{i-1} - inner radius of the (i) ring, $p_{i-1,n+1}$ - pressure between (i-1) and (i) rings produced by assembling of (n+1) ring.

The last two rings, from the current assembly, are shrink-fit with a shrinkage interference δ_{Ri}. If p_{fn} is the shrinkage pressure produced by (n+1) ring assembling from the continuity equations one obtains [7]:

$$\delta_{R_n}=\frac{1-\nu_{n+1}}{E_{n+1}}\frac{p_{fn}\cdot R^2{}_n}{R^2{}_{n+1}-R^2{}_n}R_n+\frac{1+\nu_{n+1}}{E_{n+1}}\frac{p_{fn}\cdot R^2{}_{n+1}}{R^2{}_{n+1}-R^2{}_n}R_n-$$
$$-\frac{1-\nu_n}{E_n}\frac{p_{n-1,n+1}\cdot R^2{}_{n-1}-p_{fn}\cdot R^2{}_n}{R^2{}_n-R^2{}_{n-1}}R_n-\frac{1+\nu_n}{E_n}\frac{(p_{n-1,n+1}-p_{fn})\cdot R^2{}_{n-1}}{R^2{}_n-R^2{}_{n-1}}R_n \quad (3)$$

In the next system of equation (6) we use the following notations:

$$K(n)=-\frac{2R_nR_{n-1}}{E_n(R^2{}_n-R^2{}_{n-1})} \quad (4)$$

$$H(n)=R_{n-1}\left[\frac{1}{E_n}\left(\frac{R^2{}_n+R^2{}_{n-1}}{R^2{}_n-R^2{}_{n-1}}+\nu_n\right)+\frac{1}{E_{n-1}}\left(\frac{R^2{}_{n-1}+R^2{}_{n-2}}{R^2{}_{n-1}-R^2{}_{n-2}}-\nu_n\right)\right] \quad (5)$$

Using relations (2) and (3), for multilayer construction made from (n+1) different rings (with different materials properties), the following system with (n-1) equations is obtained.

$$\begin{bmatrix} 0 & 0 & . & . & R_{n-1}K(n) & H(n+1) \\ 0 & 0 & . & . & H(n) & R_nK(n) \\ 0 & 0 & . & . & R_{n-1}K(n-1) & 0 \\ . & . & . & . & . & . \\ 0 & R_2K(3) & . & . & 0 & 0 \\ R_1K(2) & H(3) & . & . & 0 & 0 \\ H(2) & R_2K(2) & . & . & 0 & 0 \end{bmatrix}\begin{bmatrix} p_{1,n+1} \\ p_{2,n+1} \\ p_{3,n+1} \\ . \\ p_{n-2,n+1} \\ p_{n-1,n+1} \\ p_{fn} \end{bmatrix}=\begin{bmatrix} \delta_{R_n} \\ 0 \\ 0 \\ . \\ 0 \\ 0 \\ 0 \end{bmatrix} \quad (6)$$

For (n+1) rings, (n) systems are written.

Consequently, p_M, the pressure on the external surface of the die (the inner surface of the ring number 1 in fig. 5) is:

$$p_M = p_{f2} + p_{1,3} + p_{1,4} + ... + p_{1,n+1}, \tag{7}$$

where p_{f2} is the pressure between ring (1) and ring (2) represented, in the system (6) by n=2; $p_{1,3}$ the pressure between ring (1) and ring (2) represented, in the system (6) by n=3;…., and $p_{1,n+1}$ the pressure between ring (1) and ring (2) represented, in system (6) by n=n+1.

3. Experiments

3.1. Experimental set-up and used procedures

In the following experiments three grades of WC-Co alloy, four dimensions of the inner diameter of the die and 3 grades of steel are used. The number of steel rings used in these constructions is between 6 and 10 [8-10].

The experiments are made using the following procedure:

- all the components are measured with instruments having 0,002 mm tolerance;
- using the dimensions of the die and binding rings and the system (6) all interface pressures and the inner diameter of the die after assembly of each ring are computed. With the same system, the dimension of the last ring after assembly in every step of the procedure is also calculated;
- the die and the first ring are assembled by heating (the first steel ring is heated to 450°C and assembled with the die);
- inner diameter of the die and the external diameter of the steel ring number 1 are measured;
- the steel ring number 2 is pressed on the assembly die-ring number 1. In this case the contact surfaces are conical (1°-1.5°);
- inner diameter of the die and the external diameter of steel ring number 2 are measured; for rings an indexometer with 0,002 mm tolerance is used;
- the operations are repeated for all steel rings.

The levels of the stress in all components are very high; for this reason, the safety procedures are essential in the experimental process.

3.2. Physical-mechanical properties of the used materials

In the construction of the assembly die-binding rings different materials are used:

a) dies made from three grades of WC-Co alloys. The physical-mechanical properties are presented in table 1 and 2.

Table 1

Physical properties of the WC-Co alloys used in tested high-pressure reactors construction [11-13]

Type	Composition % by weight	Carbide grain size (μm)	Density (10^{-3} kg/m^3)	Hardness (HV30)	Magnetic saturation (e.m.u.)	Coercivity (Oe)
'P-R'	9 Co-91Wc	1,5	14,60	1300	165	160
'B'	9 Co-91Wc	2,7	14,64	1350	160	130
'S'	11Co-89Wc	2,7	14,40	1250	200	120

Table 2

Mechanical properties of the WC-Co alloys used in tested high-pressure reactors construction [11-13]

Type	Young Modulus (GPa)	Compressive Strength (MPa)	Transversal Rupture Strength (MPa)
'P-R'	590	-	-
'B'	596	4250	3300
'S'	575	4800	2700

b) rings made from steels.

Only two type of steel for rings are used. Because it is necessary to measure the external diameter of the last ring after assembly, the safety ring is not used. The mechanical properties of the used steel are presented in table 3.

Table 3

Mechanical properties of the steels alloys used in tested high-pressure reactors construction.

Type of steel	Tensile strength (GPa	0,2% proof strength (GPa)	Z (%)	Hardness (HRC)	Observations
SIS 2242	2,05	1,65	40	54	First ring only
SIS 2550	1,47	1,30	45	45	

3.3. Measurements during the assembly process

During the assembly process the inner diameter of die after assembly of every ring is measured. Because four dimensions for the die are used (the dies have the same geometry but different dimensions) strain at the inner diameter (theoretical and actual) is computed. In fig. 6, the correlation between theoretical and actual strain is represented.

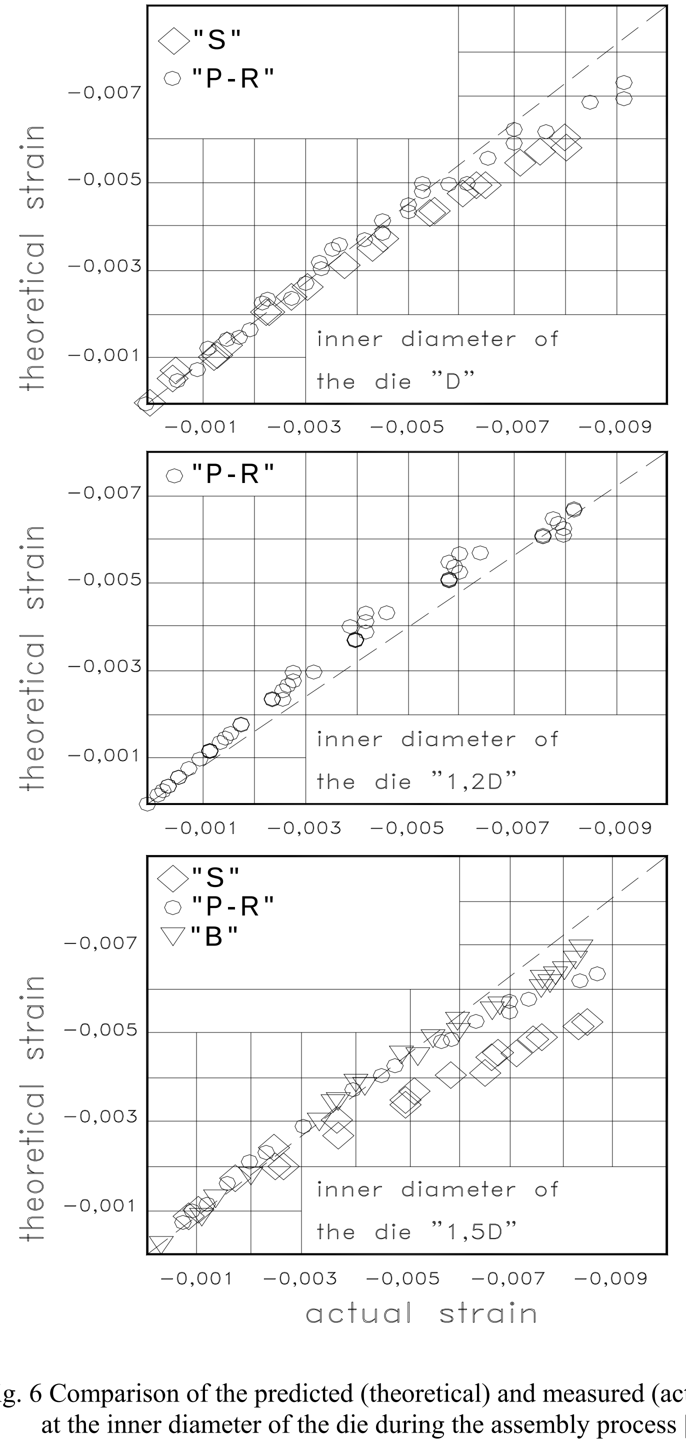

Fig. 6 Comparison of the predicted (theoretical) and measured (actual) strains at the inner diameter of the die during the assembly process [8, 9].

From the datas shown in figure 6, correlations between the theoretical and actual strain are determinate [5,6] “Table Curve Windows” software using:

$$\varepsilon_{th} = \frac{a + c \cdot \varepsilon_a}{1 + b \cdot \varepsilon_a}, \tag{8}$$

where ε_{th} is the theoretical strain, ε_a - actual strain, a, b, c - constants.

The constants from equation 8 are represented in table 4.

Table 4

The experimental constants used in equation (8)

Type of the die / Inner diameter of the die	a	b	c
“P-R” / ”D”	$4{,}9814 \cdot 10^{-5}$	28,6496	0,9909
“S” / ”D”	$7{,}5291 \cdot 10^{-5}$	29,4204	0,8997
“P-R” / ”1,2D”	$4{,}9632 \cdot 10^{-5}$	53,3740	1,1527
“P-R” / ”1,5D”	$4{,}4829 \cdot 10^{-5}$	72,3442	1,1976
“B” / ”1,5D”	$-2{,}8914 \cdot 10^{-5}$	40,3120	1,0766
“”S” / ”1,5D”	$26{,}6590 \cdot 10^{-5}$	40,2646	0,7964

In fig. 6 is shown an important plastic deformation in room temperature conditions. This plastic deformation is the difference between theoretical strain (elastic behavior equations) and actual strain. The dies are manufactured from WC-Co alloy, material with a very high Young modulus. WC-Co alloys has a fragile behavior in room temperature and pressure conditions. Bridgmann and other scientist, have experimental determinated WC-Co sample ductile behaviors in high hydrostatic conditions (hydrostatic pressure grater than 2-3GPa) [6, 14].

3.4. Measurements during the assembling and disassembling process

Correlations between theoretical and actual strains at the inner diameter of the die during the assembly and disassembly process are represented in figure 7 [8-10]. Experiments are made using two grades of WC-Co alloy and two dies with different dimensions of the inner diameter of the die.

The behavior of the dies with different dimensions but made with the same material is similar. The actual and theoretical strains are closed.

If dies made from different grade of WC-Co alloy are used, the die made from the WC-Co alloy with the lowest Young modulus has the greatest plastic deformation.

In the disassembling process a linear correlation, but with different slopes is present.

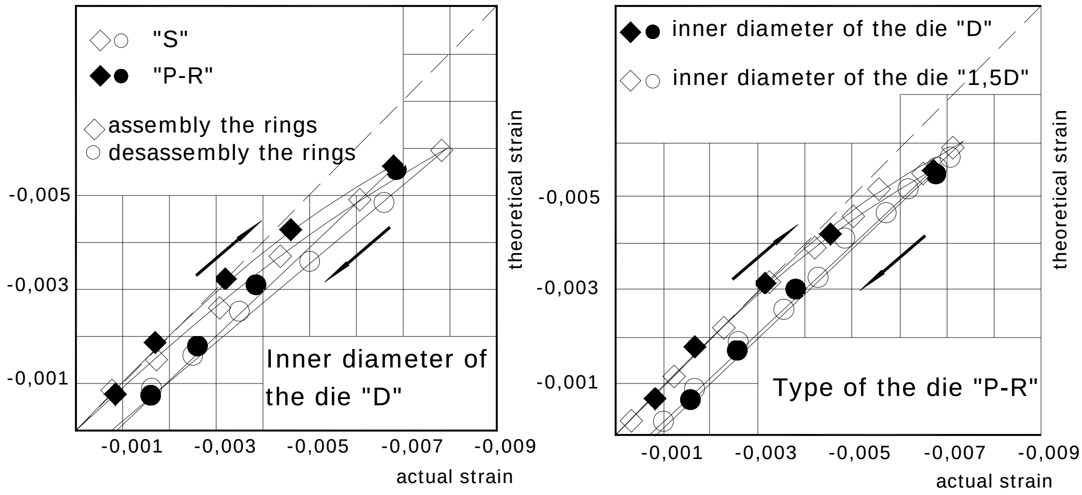

Fig. 7. Correlation between theoretical and actual strain at the inner diameter of the die during the assembly and disassembly process [8-10].

4. Conclusion

It is possible to use a simple and efficient system of linear equations in designing a multilayer construction and correct the theoretical value with a computed coefficient. In this case the procedure used in construction of a multi-rings system made from different materials is:

c) the coefficients used in equation 8 are determinated;
d) all the components are measured with devices having 0,002 mm tolerances;
e) using the system of equations (6) the theoretical values of the strain are computed at the inner diameter of the die;
f) using equation 8 or the diagrams from figure 6 the probable actual value is computed. If this value is not an optimal value, some rings (most easiest is ring number 1 after the assembly process with the die) are machined after computing the values of diameters;
g) the assembly process is started.

Diagrams from figs. 6, 7 only for the same geometries and the same material properties like the geometries and materials used in experiments are correct. For other geometries and material properties new experiments are necessary.

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