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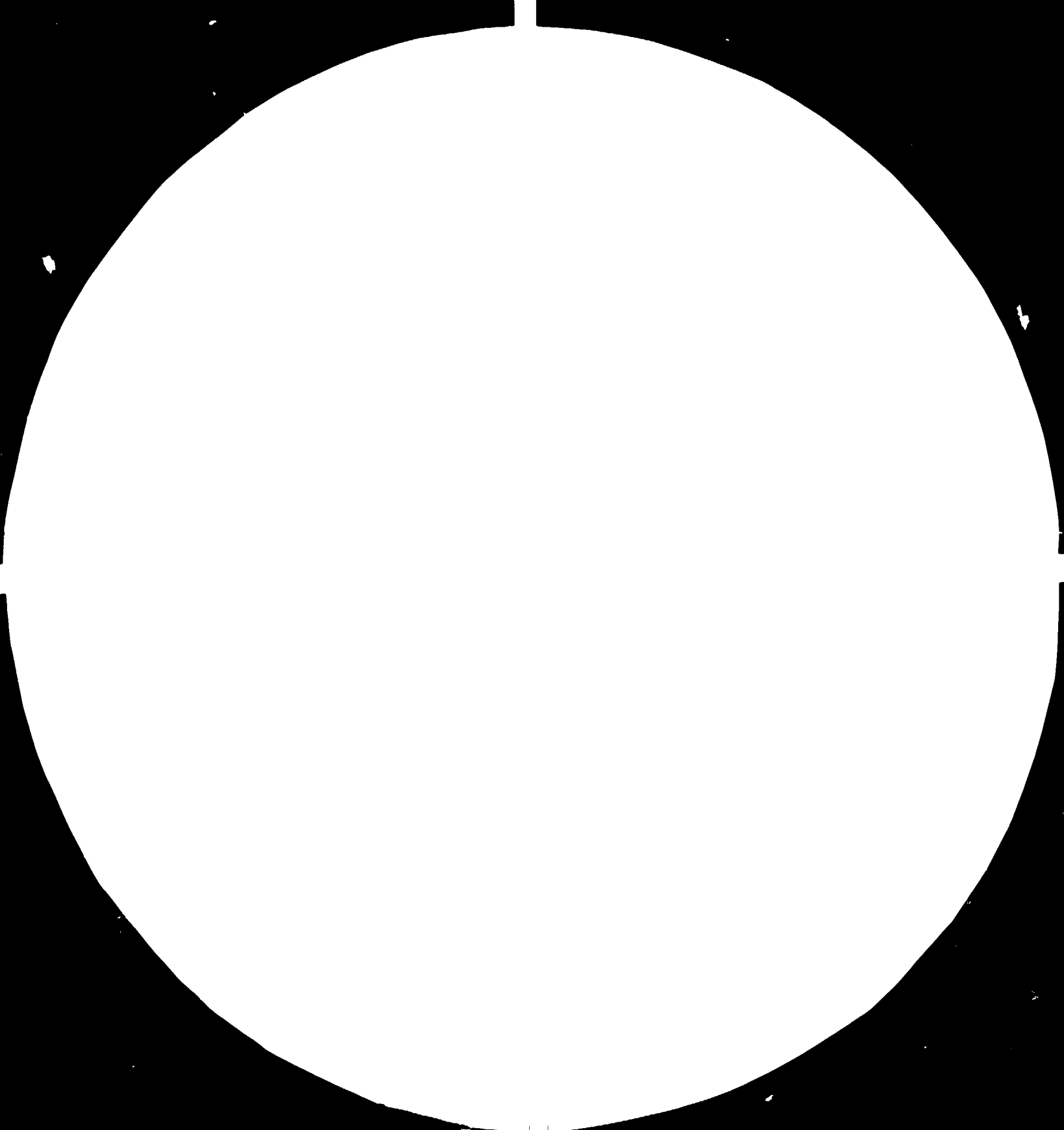
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Karl Fischer

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INDIA.

REPORT on start-up of pilot polymerization plant for Polyamide 6, 66 and Polyester

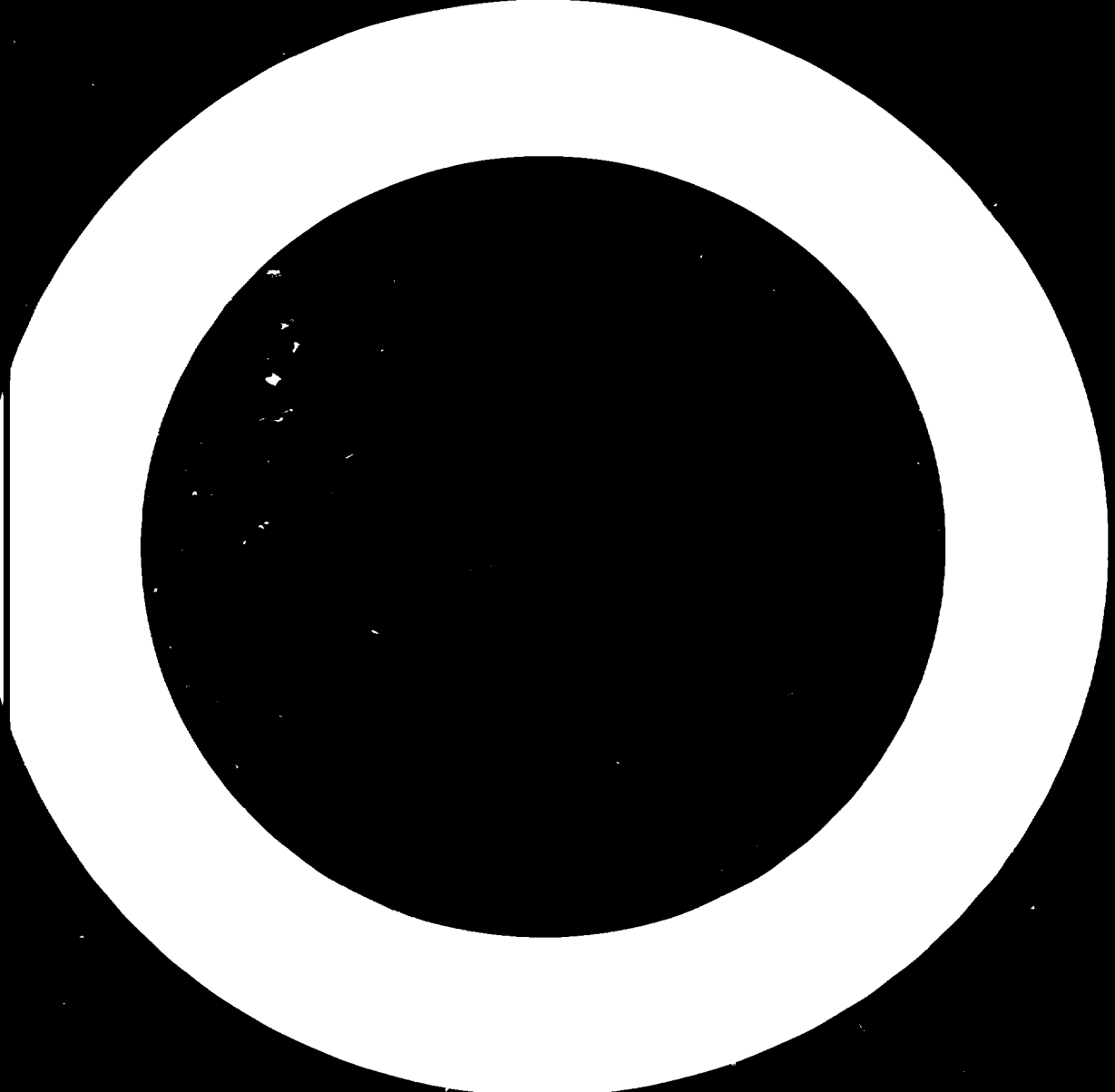
at SASMIRA during 17th April to 10th May 1983

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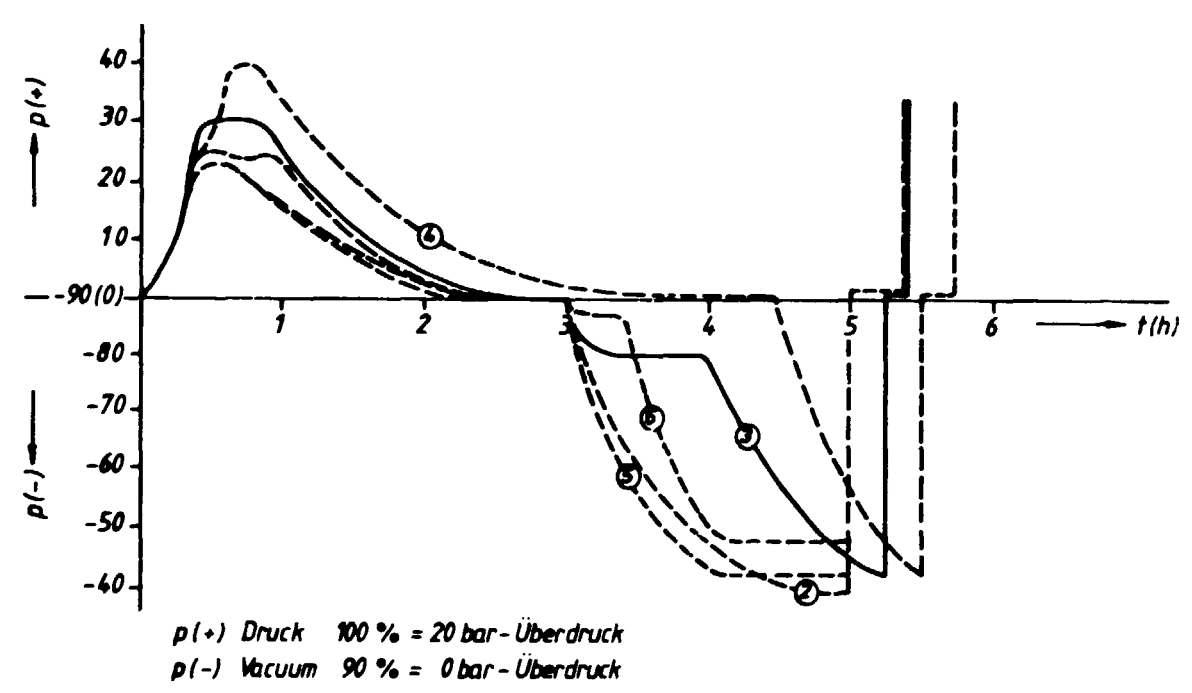
2. Start-up of plant

Because of the described manipulations partly done not very professionally, the already checked function conditions of measuring and control equipment had to be regained. Therefore, a lot of electrical work had to be done, which was not known when the agreement for the delegation and stay of KF-personnel (UNIDO CONTRACT No. 82/40) was signed, i.e. KARL FISCHER carried out a lot of unpaid work and services.

These services were done by Mr. Nietsch and Mr. Wagner from KF in 3 weeks. Only then, work could be restarted at that condition of 18th September 1981, when function tests were stopped. All heating systems were heated to the necessary temperature to produce PA 6 and process was simulated as far as possible in the autoclave. Then it was found out, that the computer could not carry out repeating operations, e.g. valve opening/closing in one program sequence. Therefore, the plant was manually started on 30th April. Plant function for the production of PA 6 was demonstrated during 6 subsequent days with 1 batch, each.

Process results and technological parameters are to be seen from the following figure and table.

Prozeß/Zeit - Diagramm



Analyse durch	CH ₃ COOH i. Lactam		rel. Viskosität		TiO ₂ -Gehalt		Extrakt	
	SASMIRA	KFI	SASMIRA	KFI	SASMIRA	KFI	SASMIRA	KFI
Charge Nr. 1	0,137 %		2,74				10,049%	
Charge Nr. 2	0,138 %		2,64		0,183 %		9,225%	
Charge Nr. 3	0,146 %		2,69		0,181 %		8,27 %	
Charge Nr. 4	0,117 %		2,55		0,169 %		8,87 %	
Charge Nr. 5	0,120 %		2,74		0,213 %		8,14 %	
Charge Nr. 6	0,146 %		2,86		0,146 %		12,32 %	

Abb./Fig. 1

Starting data

Lactam quantity:	150	kg
TiO ₂ -suspension (ca. 4,4 %):	6473	g
CH ₃ COOH quantity:	210	g
Autoclave temperature (heating):	272	°C
Lactam temperature after dosing into autoclave:	128	°C
Polymere-temperature: appr.	265	°C

The deviating results are based on the following:

1. Measuring range of balance ended at 100 g. Therefore, 210 g had to be weighed in several portions. Furthermore, calibration weight were no longer found after batch 3. Correction of CH₃COOH in lactam was not possible, as analysis results were received only after 3 hours.
2. Weighing of TiO₂-suspension could not be done properly, as it has to be carried out with balance of TiO₂-adjusting tank (mounted). Due to the tank connection with several PVC-tubes, the weighing system is no longer sensitive for g-measuring. The balance planned by KF has been placed in the spinning section.
3. Different results of post-polymerization vacuum-phase, due to wrong function of vacuum control and disturbances in steam system.

Hereunder, some comments to Fig.1:

Pressure phase is not controlled. Pressure and time is determined by means of a nozzle by evaporation of water fed in. Therefore, this water quantity is important for the operation phase. Should the pressure phase (graph) be different, e.g. by nozzle plugging as in batch 4 or by wrong weights, normal condition is regained or corrected by opening a stand-by nozzle with equal or higher bores.

The plugged nozzle has then to be unscrewed (exchanged) from control valve and cleaned. In order to demonstrate such a process deviation, nozzle at batch 4 was only exchanged at the beginning of vacuum phase.

Vacuum-phase is principally described above, i.e. evacuation of autoclave is done by pumping off the condensable and non-condensable gases through a nozzle. Necessary vacuum before nozzle is controlled by feeding saturated steam into vacuum system.

After the process, all 6 charges were discharged by casting device, i.e. the wires in liquid state are cooled in the casting vat and then cut to cylindrical chips. After several trials, operation with 18 wires must, however, be interrupted at batch 1, as the wires could not be cooled sufficiently at temperature conditions of 25°C (inlet). Temperature of 14°C, which is required acc. to the technical documentation, could not be reached in the following batches 2 and 3 due to temporary disturbances of steam supply. Therefore, only 9 instead of 18 wires could satisfactorily be discharged at batch 2. From batch 4 onwards, the wires were fed into the granulator within a short time and the batch could be spun out with lowest waste rate due to a water temperature of appr. 18°C. However, even with this temperature, the wires are too hot, so that modification of casting vat (cf. section modification) is necessary.

<u>Discharge capacity and parameter</u>		<u>PA 6</u>	<u>PA 6.6</u>	<u>PES</u>
wire diameter	(mm)	1,8	1,8	2,5
chips weight	(g/m)	2,9	2,9	6,5
number of wires	9 (18)	18	18	
draw-in speed	(m/min)	85 (44, 13)	96, 74	54, 27
melting quantity	(kg)	150 (143)	101	127
spinning time	(min)	67,6 (90)	20	20

() acc. to technical documentation

The chips of batches 2, 3 and 4 dosed into the movable chips tanks were then fed into the extractor and dried acc. to the process description, section 6.4/185 - 6.4/211 - 6.4/238. However, result of "residual extractables in chips" and "final moisture of chips" was not received before finishing of start-up, as "drying" had to be interrupted due to steam failure on 7th May to 9th May.

3. Conclusion after first plant operation with PA 6

The autoclave polymerization in the present condition is suitable to produce Polyamide 6, when considering the above described hints. However, it requires a high specific knowledge and responsibility from the operating personnel, as the process has to be "manually" controlled, cf. section 5. "Necessary actions for polymerization of Polyester and Polyamide 66".

The plant is only suitable for the production of Polyester and Polyamide 66, after some repairs have been made. The reasons are:

- heating of "ester interchange" and "polycondensation" at the production of Polyester cannot be operated by the temperature program acc. to 6.4/45 - ester interchange and 6.4/130 - polycondensation due to the failure of temperature program controller. A manual periodical adjustment of process temperature is principally possible and has perhaps to be done during the first start-up. It requires, however, high knowledge of process and sequences from the personnel.
- process (ester interchange) cannot be observed due to the failure of recorders and indicators.
- necessary vacuum cannot be obtained, as autoclave is untight due to the provisional repair of process valve
- batch cannot be spun out in the required time
- tumble dryer is too untight for Polyamide 66

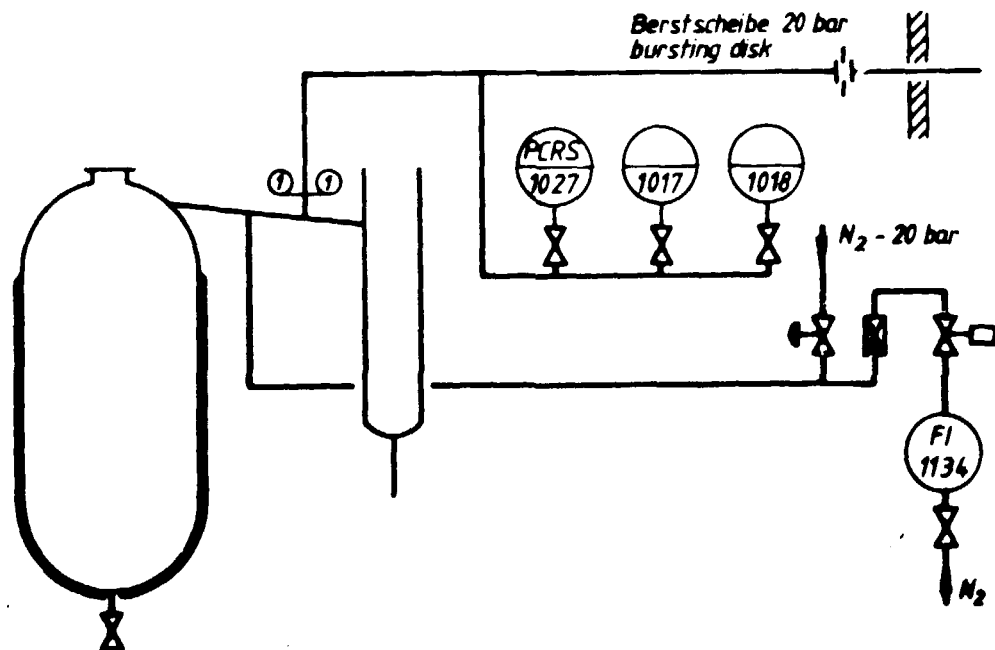
This summing up does not represent all faults, it shall only give some examples. Moreover, some modifications have to be carried out in order to arrive at a troublefree operation, especially to produce Polyester and to save energy.

4. Modifications

Polycondensation, 03.1000

It is absolutely necessary to connect the transmitter via a piping with the autoclave in order to have a proper measuring of operation pressure within the autoclave, which is a prerequisite for the release of exact controls from the computer. Process nitrogen has to be fed in via another system. By measuring and nitrogen feeding through piping DN 15, pressure within the system is built up when feeding nitrogen (20 bar) to increase pressure at the end of vacuum process phase. By this, an irratic condition is simulated to the transmitter. Acc. to Fig. 2, the transmitters PCRS 1027, 1017 and 1018 have to be connected to the autoclave exhaust line.

Abb./Fig. 2



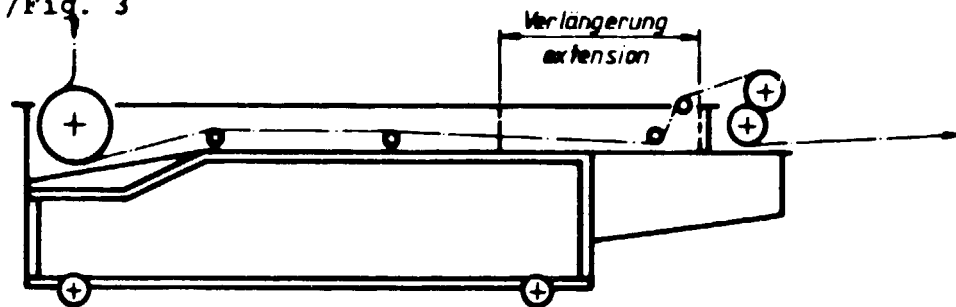
This modification can be done at site by only little alteration of piping. At first, bursting disc - which is welded in directly behind outlet of exhaust line into the connection autoclave condensator - has to be removed acc. to Fig. 2 from 1 - 1. Then, the systems are divided accordingly.

In this connection, the bursting discs of autoclave and of condensator have to be exchanged, as somebody has worked on them. This, of course, is strictly forbidden at safety devices.

Casting device, 04.1300

Precondition for the production of Polyester- and Polyamide 66-chips is that melt discharge in the autoclave will be carried out in the shortest possible time, max. 40 min. For this, drawing-off of 18 wires in melted state out of the casting head is necessary. Then they will be cooled in the casting vat so that handling and drawing into cutting device is possible. This condition, however, can only be obtained - even if cooler water is fed into casting vat - by extension of the wire water path in the vat. For this, casting vat must be enlarged by min. 1,2 m. Acc. to Fig. 3, the reverse vat wail has to be separated and a relevant U-shaped and trimed sheet plate must be incorporated, then all welded together. Take-up duo, mounted on a pad has to be screwed to the casting vat frame. This extension of the vat can be executed at site.

Abb./Fig. 3

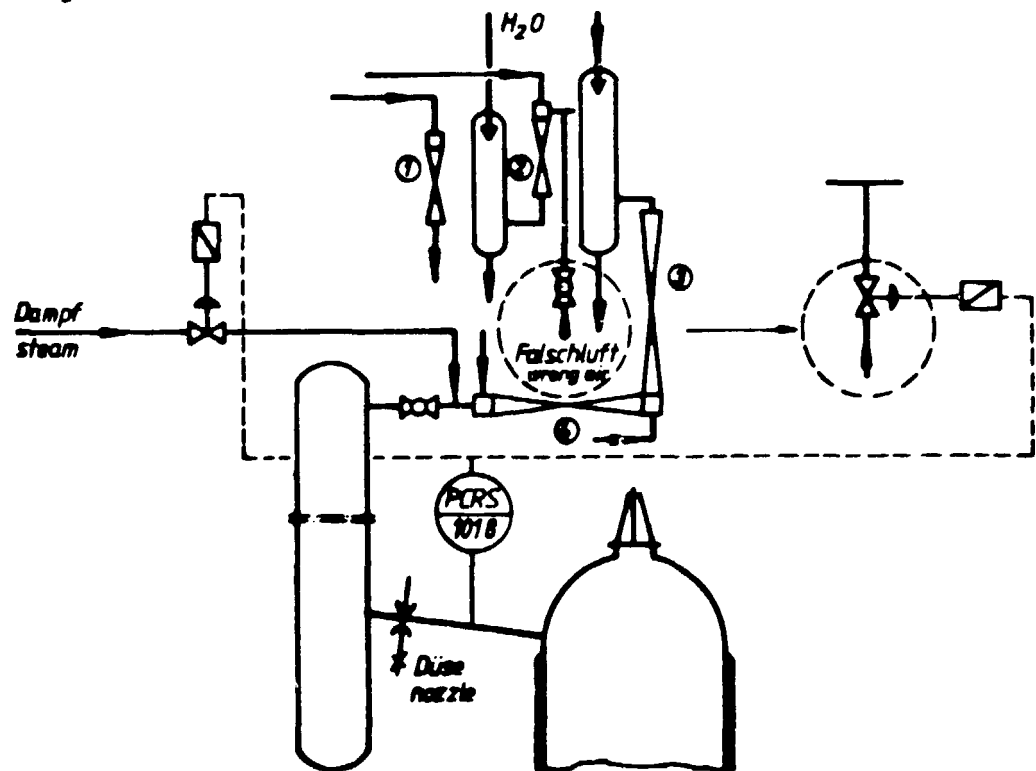


Chips cutting device has then to be moved accordingly and a new socket (higher DN) has to be welded into water reflux system.

Autoclave-vacuum control

Acc. to Fig. 4, autoclave is equipped with a 4-staged steam jet aggregate to pump EG-vapor off and to produce the necessary fine vacuum for the production of Polyester. The autoclave is evacuated to the desired pressure in this device. This high vacuum is, however, not necessary for the production of Polyamide 6 (66) so that a control system has been provided to adjust the pressure up to 300 Torr, depending on the product. This control system adjusts the desired pressure (final pressure) by feeding of by-pass gas (steam), dosed in through a bore placed in the connection flange at jet system.

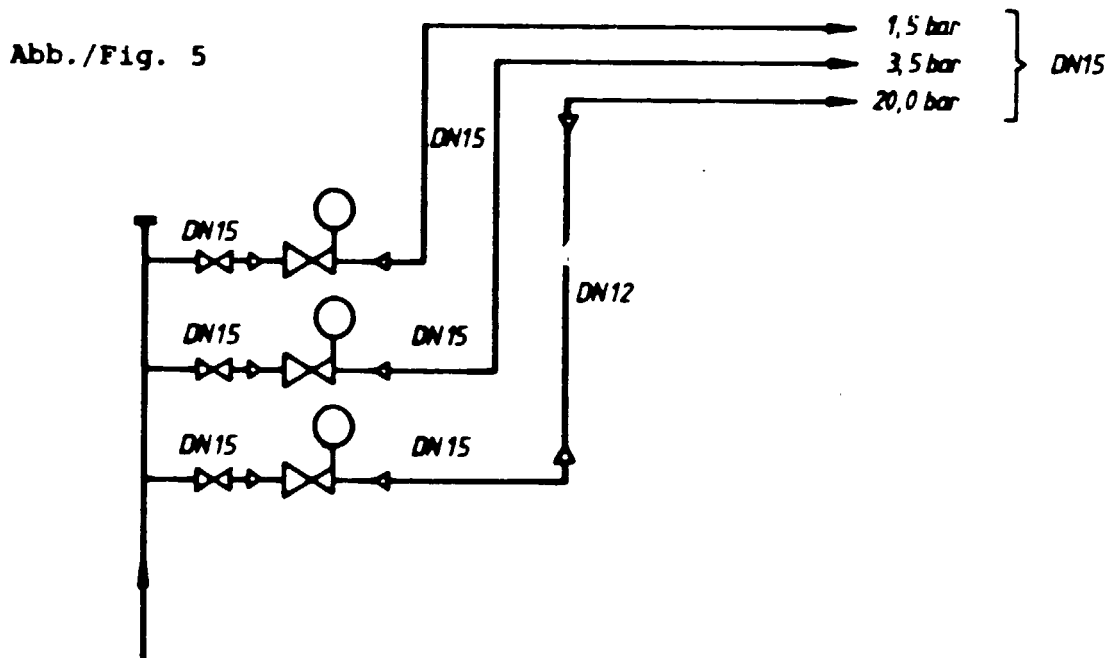
Abb./Fig. 4



As no sufficient steam could be dosed in to balance the vacuum, this design has to be improved. Therefore, during the test runs, a certain air quantity of injector 1 was sucked in by a ball cock before injector 2. In order to simplify the control system (base control load), to save saturated steam and to improve operation safety, this steam feeding system should be dismantled and the control valve should directly control the by-pass gas dosing into the system. Scope of this work is low, considering that only the control valve has to be displaced, i.e. the pneumatic controller rests where it is and only the control air piping (PVC-tube) has to be extended.

Nitrogen feeding system

Another prerequisite for quick spinning of Polyester- or Polyamide 66 melt is that autoclave can be pumped to the necessary discharge spinning pressure with nitrogen soon after having finished the process. This is at present impossible since acc. to Fig. 5 all reduction station of the 3 pressure systems 20, 3,5 and 1,5 bar are designed too small.

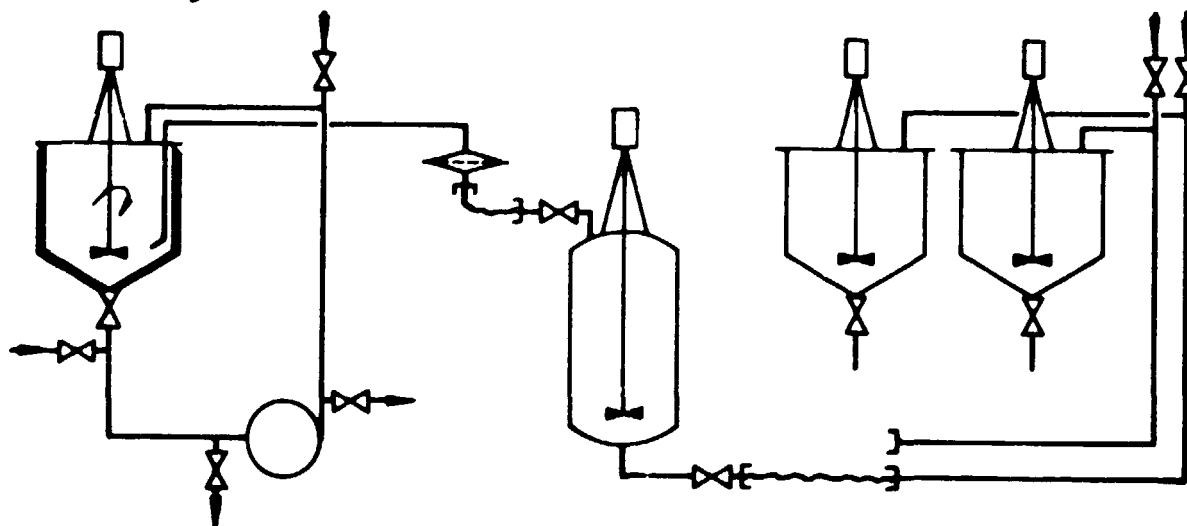


This system was outside "battery limit" and made available by SASMIRA.

Delusterant preparation

In order to receive exact and stable suspensions and so to prevent plugging, it is necessary to discharge and to clean all piping within the plant after having circulated the suspension. According to Fig. 6, subsequent valves have to be welded into the fixed piping and the flexible piping have to be equipped with quick acting couplings. In order to clean the floor which may be spilled with suspension, a water point with tube connection is to be installed into the separate room of TiO_2 -preparation plant.

Abb./Fig. 6



Stored process computer

In order to produce different Polyamide 6- 66, and Polyester chips, the plant is equipped with a stored process computer. The necessary process operations are to be fed into the programming units and the individual motors and valves are set by the computer.

During start-up of this computer, it was found out that the same valve cannot be operated several times in one process sequence.

Therefore, the manufacturer Messrs. Philips were asked to check this problem.

In the meantime it was found that the computer allows for the required operations by only slight modifications to hardware, but completely new software which can be made by KARL FISCHER.

Additional general services

In addition to the above described modifications, the following work is necessary:

1. Exchange of filter 02.1700 between ester interchanger 02.1100 and autoclave 03.1100. This monomer filter is too small in its filter surface and the wire mesh is too fine for the production of Polyester. Due to this, the filter cartridge has to be exchanged and cleaned after each batch. Furthermore, considerable TiO_2 -particles are hold back because of the cartridge fineness. These particles are no longer considered as agglomerates. When exchanging this filter, cartridge may be used for the raw material filter 01.1400 between melter 01.1100 and ester interchanger 02.1100.
2. Repair of process control valve 1024.15 in connection line autoclave 03.1100/ vapor condensor 03.3100. This valve which controls the pressure and vacuum phase, had to be repaired during start-up but only provisionally due to missing spare parts. In this condition, it is not suitable for a continuous operation, especially for the production of Polyester. The valve has to be checked completely and revised.
3. Dosing device 03.4100 (ester interchanger) and 03.4200 (autoclave) are to be equipped with exhaust valves. By this, air escapes quicker during dosing of catalyst and reflux of dosed-in liquid is omitted. Item 03.4100 has already been exchanged.
4. Spindle and seat of nitrogen valve 1020.15 has to be grinded anew for control of spinning pressure. This valve is so untight that nitrogen pressure within autoclave increases slowly during spinning and has then possibly to be discharged. Due to this untightness, nitrogen flows permanently through the autoclave resulting in a permanent nitrogen loss. Such untightness has been observed also at other valves so that all valves have to be checked and possibly to be grinded, too.

5. Exchange of several valve inserts in control valve for control improvement.
6. Exchange of simmer rings in tumble dryer.
7. Some general service work.

Summary

All modifications listed above can be considered as the normal result at start-up of a plant. Due to the disastrous situation of the plant in the electrical part at the beginning and the limited time, these work could not be made immediately. Some repairs have still to be made due to long idle time of the plant and its partly set into operation before 17th April 1983. This holds especially for valves, dryer, autoclave untightness and several electrical and control components which must be renewed.

5. Necessary actions for polymerization of Polyester and Polyamide 66

During start-up, it was proved that the plant designed for computer control, is able to produce Polyamide 6, although the process stages "ester interchange/precondensation" and "polycondensation" have been operated manually. This manual operation required detailed knowledge of all apparatus and plant design. It was not possible to train the SASMIRA personnel in one week (6 batches) sufficiently enough. SASMIRA intends to operate the plant for the production of non-delusterant Polyamide 6 after our return home. This report refers only to that situation, when we left, regarding the necessary modifications and required spare and wear parts. All these parts have to be bought, otherwise Polyester and Polyamide 66 cannot be run.

Actual plant personnel has to be increased, whereas the two electricians should be able to run their jobs.

Laboratory needs to be organized in order to have test results in required time and accuracy, which is of paramount importance at polymerization of Polyester.

Some of the modifications can be carried out by SASMIRA's personnel. Delegation of KARL FISCHER personnel should be made in two steps:

- inspection 3 weeks prior to the requested delegation by 2 technicians/engineers
- delegation of start-up team after the inspection has proved plant to be ready for start-up.

20th May/ 8th June 1983

TP/Pk-bb

Annex 1

Measurement and results of the start-up carried out by SASMIRA personnel on occasion of the "12th Technological Conference"

The following modifications were found by Mr. Wagner of KARL FISCHER when he started his work on 4th April 1983 under the supervision of Mr. Gokhale - SASMIRA:

Cabinet 15 (Procon)

connection strip 15 L1: connections cut off, cable partly wired together

dito 15L3 bridge cut off, former connections removed, not marked

dito 15L4: alarms (in- and outlets of Procon) removed or cut off

others: in cable assembly wrong lines wired with each other

Cabinet 12 (ester interchanger)

New cable laid for current supply of 4 switching devices, resulting in wrong voltage (all 4 device damaged)

Connections of safety barriers overwound during adjustment or cutting off.

Cabinet 16 (heating)

Manipulation carried out at THYROTAKT of second heating circuit

Cabinet 18 (motor control)

New cable laid and pinched, old connections removed and not marked.

Within the plant (at site)

Manometer (5) and resistance thermometer (4) of heating circuits cut off.

Motors equipped with switches for direct connection

Several limit switches cut off from valves or dismantled

Motor 03.1:00 (agitator-autoclave) blown out

The following listed devices are damaged and have to be repaired or bought anew:

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LIST OF THE DAMAGED PARTS

- 1) Three nos. of Programmers
- 2) Three nos. of Temperature Recorders. [Mechanic System]
- 3) One no. of Pressure Transmitter.
- 4) ~~Three~~ ^{Four} nos. of Temperature Transmitters cards.
- 5) Safety controlled 'Switch ON' Units → 4 NO^s.
- 6) ~~Eight~~ ^{Eight} nos. of safety Barriers Unit NO. WE 74/E x 2.
- 7) Bearing NO. I. [9404 206 64001]
Five nos. of Safety Barriers
- 8) Bearing NO. R. [9404 206 52001]
2 NO^s of [1st Block] Contactor.
- 9) Three nos. of Overload Relays.
- 10) Viscosity Meter. (1 NO) [Contacts not working]
- 11) Vacuum Meters (2 NO^s)
- 12) One no. of Contact Unit.
- 13) Two nos. of vacuum sensors.
- 14) Vacuum Transmitter and safety arrangement [Over
- 15)

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