IX SCALE CHANGE OF A PROCESS

In all of the experimental disciplines, experimentation is done on as small a scale as is practical for the sake of both convenience and economy. If a process is to be developed to a commercial scale, the scale enlargement practically always poses problems. The problems of scaling are related to the physical and engineering aspects of a process. Since fiber formation processes are almost always entirely physical, and very complex at that, the scaling often poses great difficulties. The most general advice based on long experience teaches that the more time spent in a library, the less time is needed in the laboratory, the more time spent in the laboratory the less time is needed to scale the process, the more time spent on scaling the better the machine design and the smoother its start up.

After the experiments on a small scale are completed, the product which possesses the same properties must be obtained in larger volumes. Manufacturing equipment must be enlarged, in comparison with the experimental machines, in such a way as to assure that the theoretically essential variables will remain unchanged. In chemical engineering, some more complicated cases are divided into several steps of scaling-up, which may be advantageous. It may happen that a perfect reproducibility of the product properties in not attainable, thus it is the task of engineering to limit these bad cases to a minimum.

Generally, in the fiber producing industry, scaling of the fiber formation processes is considered to be exceedingly difficult. It appears that this is to a large extent due to the fact that scaling of fiber formation may well be one of the most misunderstood tasks of its kind. The problem of scaling fiber formation processes in any broader general sense has been treated earlier only by this author.[1] In contrast to the majority of other processes utilized by chemical industry, fiber formation does not involve chemical changes, with those few exceptions involving wet processes. A chemical process may be accelerated, or slowed down by change of temperature or pressure; the product remains essentially the same. Perhaps some of the impurities resulting from side reactions may change. In chemistry of low molecular mass compounds the impurities may be removed. In processes involving macromolecules, such a purification is practically impossible.

In the majority of physical processes, especially in those involving macromolecules, time scale cannot be changed, and this is also the case with the temperature of a process. Crystallization is a physical process whose rate depends on temperature, but along with the changes of temperature follow qualitative changes in the crystal structure, most conspicuously changes of the melting point. Similar considerations are also valid in regard to the rheological properties; time is important to relaxation and retardation processes, and the relaxation times depend on temperature.

In summary, if we want to reproduce a process in a different scale, the time and temperature profiles cannot be changed. At this point we meet another limitation: the time scale in fiber formation depends on filament diameter. Thus, for a process to be unchanged, the profile of diameter attenuation also must remain

unchanged to prevent alteration of the time scale. If these conditions are not met then everything else, like velocity of extrusion, temperature, etc., must be changed to reach an equivalency in the rates of the unit processes involved. The problems become difficult indeed.

Parenthetically, one may consider the necessary degree of constancy or the sensitivity, or tolerance of the process. This certainly varies with polymers and by no means least importantly, with the demand on the quality and reproducibility of the properties for any given product. However, such decisions to a large extent reach beyond the boundaries of technology.

To begin with, one must realize that the enlarging of the scale of a fiber making operation may be achieved truly only in one way: by enlarging the number of filaments produced in one position of a machine with identical geometry. All other ways seek some equivalency which is not a true scale-up. There is more than one way in seeking an equivalency for the processes, some of the ways may be easier than others.

The task of changing the fiber titer while all other properties remain unchanged, may also be treated as changing of scale.

IX.1 Changing the Number of Filaments

If the operation of changing the number of filaments is to be a true change of scale with all fiber properties unchanged, the machine geometry must be the same and all the technological variables, except flow of cooling media, must remain unchanged. Similarly unchanged must remain the technological variables of the drawing operation.

The only change introduced in the process is an increase of the number of filaments per position, and the resulting increased volume of polymer flow per position. A larger volume of polymer carries a larger amount of heat to be removed from the tow, but the heat removed from an individual filament should remain unchanged along the entire profile. The magnitude of the problem depends on the magnitude of the scaling factor, but after consulting the section on heat transfer the task will not be found easy.

The task may be approached by increasing the velocity (volume) of the cooling medium used, or by decreasing the medium temperature, or both. The limitations on the medium flow intensity are related primarily to the drag forces exerted on the filaments, and this is particularly unpleasant in case of cross flow. Excessive bellowing of the spinline does change somewhat the profile of forces acting on the filament. An angle between medium flow and filament other than ninety degrees causes the formation of an axial force component. In the lower segment, the component is directed against the drawing force, and in the top segment it is increases the drawing force. At higher gas velocities, there is also a threat of filament vibrations which might lead to fiber nonuniformities.

Lowering of the medium temperature is a much more appealing proposition. When the scaling factor is large, the lowering of the temperature may be limited by

economy since cooling energy is expensive. Very cold air also causes troublesome water condensation. Thus, this remedy also has its limitations. Some advice: when developing a process in small scale do not use very cold air, leave yourself a safety margin for the future scale up.

The number of capillaries is increased mostly in proportion to the surface of the spinnerette plate. In cases of circular spinnerettes, this is proportionally to the square of the spinnerette diameter. The access of quenching air to the filaments, irrespective of the type of quench or coagulation medium, is proportional to the spinnerette diameter. Consequently, to provide a comparable cooling effect, the air flow must be largely redesigned taking all the above features into account. An enlarged spinnerette may be designed in such a way that the diameter will be scaled up rather than the surface area. Such a possibility represents a relatively convenient solution in respect to the scaling of quench or drying. Nevertheless, such a convenience may be expensive in terms of other consequences, the acuteness of which increases with the increase of the scaling modulus.

When the scaling modulus of the spinnerette diameter is chosen, then the force acting on the spinnerette plate during extrusion grows in quadratic proportion to the modulus by which the diameter has been increased. In operations of multifilament formation, the force acting on the spinnerette surface may reach a magnitude that is difficult to cope with. Another consequence of such scaling would be an increase in fiber-to-fiber nonuniformity between the first and last rows of filaments in the path of the air. In such a situation a remedy may be changing the spinnerette plate from circular to rectangular. The latter may offer the possibility of retaining a reasonably similar number of filament rows and expanding the length of the plate in proportion to the increased number of filaments. Such a solution has the additional benefit that the ratio of the circumference to the area is larger for rectangles than for circles. This is certainly helpful in designing a sufficiently strong, buckling resistant mounting of the spinnerette plate. Rectangular spinnerettes certainly represent a much superior solution over large circular spinnerettes with quench air introduced into the center of the filament bundle.

Rectangular spinnerettes may be equally well suited for cross flow as for cocurrent air flow. Switching from circular to rectangular spinnerettes may also be understood as a measure to limit the excessively high air velocities which would be needed for circular spinnerettes while simultaneously maintaining the fiber-to-fiber differences within technologically reasonable limits.

In the drawing zone, large amounts of heat are developed within a small space. The degree of spatial limitation depends on how well the necking is localized. Assuming a drawing system with the necking localized within very narrow limits, the problem may be described in the following comparative way: For 1 to about 100 filament lines, the zone of drawing must be heated. The amount of heat needed decreases with an increasing number of filaments. Also, when a smaller number of filaments are drawn, it is highly advisable to heat the fibers beyond the necking point. When the number of filaments reaches some 200 to 300, depending on the draw ratio, polymer, fiber titer, and method of drawing, the threadline may

require gentle cooling. In some operations where the number of filaments reaches over 1000 per position, the filaments may display a tendency to fuse.

In the case of very heavy threadlines, temperature control becomes a difficult task indeed. Here we have a conflict of interest: for the sake of high quality, the necking ought to be well localized with a good temperature control. For a very large number of filaments satisfying the two conditions is difficult. The way out of the conflict may be to spread the threadline into a wide band for easier temperature control. Not without significance is here the choice of the type of drawing device.

Although the scaling method just described may seem to be relatively uncomplicated, it should not be assumed a perfect way of achieving the goal. In performing such a scaling up, certain changes are unavoidable in the extrusion area. In reality, the increased size of the polymer delivery equipment, such as the screw melter or autoclave, longer transfer lines, etc., increases the residence time of polymer at elevated temperature and under shear. For many polymers, this may lead to changes of the fiber properties. For some polymers this may mean increased degradation or other structural changes. When changes of molecular mass are involved, corrections may be possible, but when branching or cross linking results from the prolonged heat exposure, then there exists no corrective remedy.

The necessary changes of size, and frequently of geometry, of the polymer supply system may have an influence on the shear history of the polymer with further consequences reflected in the fiber properties or in the processing characteristics.

Another unavoidable effect of scale increase is an increase of the thickness of the spinnerette plate for the sake of its mechanical strength. This leads to the necessary alterations in the geometry of the capillary entry, which in turn may have an influence on the total shear history. The outcome of so many adjustments in the flow conditions is impossible to predict *a priori* unless a very extensive examination of the polymer rheology has been performed.

IX.2 "Scaling by Equivalence"

A change of the machine geometry or any of the technological variables would not be considered a scaling-up, but rather a seeking of an *equivalent process* that will preserve a majority of the critical fiber properties. If the machine geometry is altered so that the length of the quench zone is different, then automatically a score of technological variables will change. To bring the operation back to the point where the fiber properties will be the same is practically impossible. One may adjust the independent variables so as to create a new formation environment leading to fiber properties more or less similar to those obtained in a smaller scale. There exist situations when this is the only way available to solve the scaling operation problem. Such situations are always painful, time and money consuming, and they often lead to a frustrating repetition of a majority of the original work of the original process development.

Let us consider some possibilities of changes of the process parameters. If the

polymer flow is changed by a scaling factor of m, then to preserve the same shear rate in the capillary we have

$$\dot{\gamma} = \frac{4Q}{\pi r^3} = \frac{4mQ}{\pi r_1^3} \tag{IX.1}$$

This leads to the requirement that the new capillary diameter be

$$r_1 = m^{1/3} r \tag{IX.1 a}$$

If the original capillary aspect ratio, l/d, is preserved, along with the original shear rate, then the die swell will remain unchanged. This means that the maximum diameter of the extrudate will be $2B \cdot m^{1/3} r$ and the polymer stream velocity in the die swell will increase by the same factor of $m^{1/3}$.

If we preserve the same timing, then with identical machine geometry and appropriately scaled cooling, the filament velocity at the end of quench should increase by the same factor of $m^{1/3}$. As a result of the changes of polymer flow by the factor of m, and velocity by the factor of $m^{1/3}$, the fiber titer will increase by a factor of

$$9 \cdot 10^5 \frac{mQ\rho}{m^{1/3} v} = 9 \cdot m^{2/3} \frac{Q\rho}{v}$$

Then we have a factor of $m^{2/3}$, not m. The polymer flow problems connected with the scale changing from one segment of the process to another have been noticed earlier by W. E. Fitzgerald and J. P. Craig[2].

In view of the different filament velocities, the timing and the strain profiles may be preserved only when the quench zone is expanded by a factor of $m^{1/3}$. Here it is necessary to stress that equivalent cooling means that the temperature profile must be reproduced against distance multiplied by $m^{1/3}$ if the timing is to be preserved accurately. The net drawing force, that is, the rheology force available for extension without the losses due to other forces, must be increased by a factor of $m^{2/3}$.

Thus, we have just presented a nice scaling operation to increase fiber titer. However, there are certain questions remaining: How much can the titer be increased in this way? If we have the possibility of extending the quench zone by 14.47 per cent, then we may increase titer by 31 per cent. If, for example, the titer increase is from 10 dTx to 13.1 dTx, this is significant. If, however, there is a question of changing titer by the same percentage but for finer fibers, say from 2 dTx to 2.62 dTx, it is not very significant. If a greater increase is needed, then another question arises: How big a flexibility in expanding the quench zone should a machine have? Usually, increased flexibility means increased investment costs.

Occasionally one may find in the literature papers describing the attempts to intensify extrusion with preservation of the spun fiber diameter. To evaluate such a situation, one may conduct considerations similar to those described above. In this case, the filament velocity at the end of quench zone would have to be larger by the scaling factor m. In such a case, however, the extension would increase $\lambda = m/m^{1/3} = m^{2/3}$. If the length of the quench zone were increased by $m^{2/3}$,

then the original temperature profile *versus* distance may possibly be reproduced. The problems begin when we consider the attenuation profile and the connected time scale.

The present knowledge in the area of creep (extension) is insufficient to solve the problem theoretically. To successfully accomplish the task, one needs to find some way to manipulate the stress - temperature - time profiles in order to obtain a similar structure formation. This kind of work is no longer a scaling, but a clear reworking of the most difficult segment of the process *"from scratch"*.

Another way of solving such a problem may be through redesigning extrusion to the point that the minimum filament velocity (in the die swell) will be increased by the factor of m, and thus final fiber velocity, $v_{f_1} = m \cdot v_f$, will be increased by the same factor. The cooling must be redesigned to preserve the original temperature profile over the quench length, scaled also by m to preserve both the timing and the degree of extension, λ.

The remaining problem is how to increase the minimum velocity of the filament. Velocity is in direct proportion to the volume of the polymer flowing with an unchanged diameter, but shear rate increases also by the same factor m. The latter results in an increase of die swell. The solution of this will depend on the polymer in question. Sometimes extended capillary length may help to lower again the increased die swell, provided there is room to increase the resulting higher extrusion pressure. Decrease of the capillary diameter is in such cases counterproductive as the velocity is affected by the square of the diameter and shear rate by the diameter to the third power, thus the die swell may be even larger. Increase of the extrusion temperature may help in reducing the die swell, other factors permitting. On the other hand, an increase of temperature would aggravate the effort to redesign the cooling to preserve the temperature profile.

In each of the cases presented in this section, the length of the quench zone had to be changed, as had some profiles of the quench zone. One change had to be traded for another, not to mention the additional difficulties which may appear, such as a higher force of the higher air velocity acting on a longer filament, and others. Flexibility of the forming machines to meet the demands represents another question. For all these reasons, this section has been named *"Scaling by Equivalence"* (in quotation marks) to indicate that these methods of scaling are not exactly a scaling as it is understood in the area of chemical engineering.[3]

IX.3 Experimental Formation Machines

Successes in research and development depend to a large extent on the availability of proper equipment. The same is true of scaling operations. The notion "proper equipment" is not necessarily equivalent to "expensive equipment". In many fiber research laboratories, one may find an impressive number of experimental machines designed specifically for the performance of different tasks, for work with different polymers, and so on.

In reality, a laboratory for research on fiber formation from melt needs one

experimental machine per fifteen to twenty persons of independently working technical personnel (engineers). In fact, two to three days of well planned experiments by an engineer with the help of an operating crew of technicians (two to three) is able to produce enough data for the investigator to work on processing the data for two to three months, and sometimes longer.

A both technically and economically efficient machine ought to be built from individual elements, something like the laboratory glassware, or Lego^R blocks. Within such a system, all kinds of experiments are possible, all types of endeavors, from basic, fundamental research to fairly far reaching scaling operations.

The heaviest section of a formation machine, in the physical sense, is the extrusion section. For this reason, this section should be planned to have a fixed location on top of the area to be occupied by the machine. The section should consist of two extruders, one smaller and one larger, connected to one block containing two metering pumps, filters, and spinnerette. The two extruders offer a possibility of forming fibers from one or several filaments to some 300 or even 500 filaments. The actual size of the extruders may be adjusted to the prevailing type of work expected in the laboratory. In case a large range of filament numbers is needed, a third little extruder for the smallest number of filaments may be "wheeled in" and connected to the block when needed.

Beside the wide range of total polymer flow, such an extrusion section offers also the possibility of working with two different polymers for bicomponent fibers or some other nonstandard combinations. Naturally, the bicomponent work requires melt distribution plates for the spinnerettes and the block must be so designed to be able to accommodate them.

Underneath of the extrusion section should be located a metal frame, just like in a laboratory for organic synthesis, but it must have strength appropriate for the size and weight of the equipment. All elements needed for the experimentation and for characterization of the process may be fastened on the railing. For the purpose of fastening the various elements on the railing, a set of clamps standardized for the machine is necessary.

The quench system ought to consist of modules of possibly variable length to allow for greater flexibility in assembling any length within small increments. All types of quench may be used here, coaxial, with the easiest subdivision into modules; cross flow or cross flow with recirculated air. The last variation is highly recommended, particularly for experimental work, but also for commercial machines since it offers a better air pattern in flow through the fiber line and reduces fiber-to- fiber variability. Increased ecological friendliness of the recirculating system is not without great significance.

The quench modules of all types should have provisions for use of characterization equipment, i.e. see-through window strips on two opposing sides with a possibility to open small sections of the windows for those techniques which cannot tolerate glass obstruction.

Another provision of importance are heaters delaying quenching. These must be built in small modules, but a total length of some 75 cm might be found useful.

Such heaters must also be equipped with transparent windows.

Work with a small number of filaments requires careful regulation of quench. Extrusion into stagnant ambient air (room temperature) results in as rapid cooling as may not be easily attainable with five hundred filaments. For this reason, the same heaters may be used also to slow down the cooling, not only for a total delay. For the same purpose, all of the quench systems with air temperature higher than ambient may be used. For this kind of experimentation, the cross flow quench with air recirculation is the most suited, the most convenient, and interferes the least with the general working ambiance.

Fume removal usually needs to be separate for each quench system and quench delay, as these pieces of hardware need to cooperate. A careful design that obtains a good match is to be recommended.

The choice of transporting rollers may be a little difficult. The question is whether to use the multiple wrap system or the S-wrap system. Another question is whether to build the moduli of single rollers and set them appropriately, or to combine two rollers in one package. The single rollers represent more flexible, but also a slightly more expensive, solution – each roller has a separate motor which needs precise velocity synchronization. The roller pair packages are heavier and more difficult to realign. The single rollers, with a good mounting bracket design may be easier to use both for multiple and S-wrap. One must remember that for the S-wrap, the rollers within a pair rotate in opposite directions.

Generally, a minimum of three pairs of rollers are needed: take-up, drawing, and relaxing. If drawing is to be done in more than one step, then each additional step requires an additional pair of rollers. The number of rollers may need to be increased if there is the necessity of covering a very extensive range of velocities. Generally, velocity regulation spans a range of 1 to 10.

Depending on the aims of the experimental work, drawing, or lay- down, jets may be considered in addition to, or instead of, the rollers.

As the preferred configuration of the machine from extrusion to take-up is vertical, the drawing section may be arranged horizontally. A decision on this point may depend on the availability of sufficiently high space. The operation of such a machine requires an vertically movable platform because of the variability of quench zone length, as well as possible changes of the configuration.

It is quite obvious that depending on the expected application of the fibers, different types of devices to receive the drawn fibers may be required – a bobbin winder, staple cutter, or others.

For successful research, the process characterization possibilities are equally important as the machine itself. Basic equipment for the purpose includes:

- Infrared equipment for measurement of the fiber surface temperature (and other surfaces).
- Photographic equipment with a *telemicroscope* or a high class, very long focal length objective, which allows photography on a close distance.
- Laser Doppler anemometer (LDA) for measurements of filament and air ve-

locities (not mandatory but necessary). In absence of an LDA, a set of good anemometers.

- X-ray diffraction and low angle scattering equipment (not mandatory but very important).
- High precision fiber tension meter.

There are necessary provisions for mounting of the characterization equipment on the frame with the possibilities of the vertical movement along the threadline, and also with the access to the spinline (i. e. the windows as indicated above).

A machine for dry formation from solution may be built very much like one for the formation from melt. The extrusion section is likely to be replaced with a nitrogen pressure feeding of the metering pumps directly from the dissolution equipment. An appropriate solvent recovery system (adsorption on active carbon) is a must. The rest of the machine is almost identical to that for the formation from melt.

The wet formation from solution has the smallest possibilities for variations. The extrusion section should be substituted like in dry formation. It is important to have the coagulation baths of different (variable) lengths. The method of just moving the spinnerette closer to the take-up roller to shorten the bath does not solve the problem adequately. Control of the flow of the coagulating bath becomes difficult. Also, use of LDA may be problematic in this case. Solvent and nonsolvent recovery needs to be done according to the solvent system used.

Experiments with a new polymer start from the absolute minimum of polymer usage. Normally new polymers are in short supply. Formation experiments start from the smallest number of filaments. Quenching must be designed appropriately to increase the number of filaments while maintaining unchanged technological variables. In the case of other development work, it is equally important to experiment with the future in mind and always asking the question: Will it be possible to duplicate the results on an appropriately large scale? A good experimental machine allows far reaching increase of scale and almost automatically forces the question: How will it be when we make more filaments?

IX.4 References

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