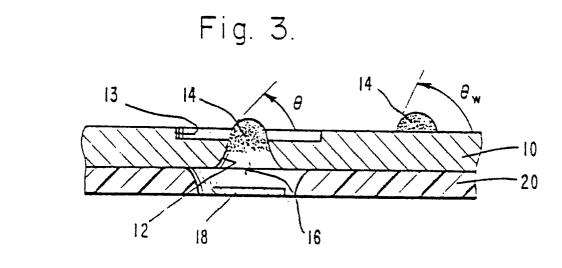
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(a) Printhead performance tuning via ink viscosity adjustment.

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The drop stability of a pen employed in an ink-jet printer and having a plurality of nozzles (12) in a nozzle plate (10) is increased by increasing the viscosity of the ink. For inks comprising water/glycols, the increase in viscosity is easily accomplished by increasing the ratio of glycol to water. In one ink, a 20 % increase in glycol concentration resulted in a 50 % increase in viscosity and a decrease in the variation of angular misdirection by 43 %.



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PRINTHEAD PERFORMANCE TUNING VIA INK VISCOSITY ADJUSTMENT

TECHNICAL FIELD

This invention relates to ink-jet printers, specifically thermal ink-jet printers, and more particularly, to a structure for substantially improving the performance of the nozzle(s) in a thermal ink-jet printhead. This improvement in stability and consistency of operation extends the firing frequency range of the nozzle and reduces cross-talk, as well as desensitizes the exterior surfaces of the jetting nozzles to their wettability state.

The design principles described herein are not limited solely to thermal ink jet applications but in fact are of value in the design of athermally excited ink-jet printheads as well.

This invention is of particular value in those circumstances of design in which other means of attaining the above-mentioned operating benefits are not available through geometry changes due to manufacturing process constraints.

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BACKGROUND ART

- When designing printheads containing a plurality of ink-ejecting nozzles in a densely packed array, it is necessary to provide some means of isolating the dynamics of any given nozzle from its neighbors, or else cross-talk will occur between the nozzles as they fire droplets of ink from elements associated with the nozzles. This cross-talk seriously degrades print quality and hence any providently designed ink-jet printhead must include some features to accomplish decoupling between the nozzles and the common ink supply plenum so that the plenum does not supply a cross-talk path between neighboring nozzles.
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Further, when an ink-jet printhead is called upon to discharge ink droplets at a very high rate, the motion of the meniscus present in each nozzle must be carefully controlled so as to prevent any oscillation or "ringing" of the meniscus caused by refill dynamics from interfering with the ejection of subsequently fired droplets. Ordinarily, the "settling time" required between firings sets a limit on the maximum repetition rate at which the nozzle can operate. If an ink droplet is fired from a nozzle too soon after the previous firing, the ringing of the meniscus modulates the quantity of ink in the second droplet out. In the case where

firing, the ringing of the meniscus modulates the quantity of ink in the second droplet out. In the case where the meniscus has "over-shot" its equilibrium position, a firing superimposed on overshoot yields an unacceptably large ejected droplet. The opposite is true if the firing is superimposed on an undershoot condition: the ejected droplet is too small and extremely fast (this is known as a spear drop). Therefore, in order to enhance the maximum printing rate of an ink-jet printhead, it is necessary to include in its design some means for reducing meniscus oscillation so as to minimize the settling time between sequential firings

of any one nozzle.

In addition to cross-talk minimization, an important objective of printhead design optimization is the control of meniscus dynamics during refill. During the overshoot phase of refill, the momentum of the fluid which has flowed into the firing chamber carries the meniscus beyond its equilibrium position. At that point where the compliance of the meniscus has halted the fluid flow, the meniscus has bulged out of the bore and appears briefly as a spherical section or "igloo" of ink projecting out of the nozzle. Within microseconds, it has retracted itself back into the nozzle bore under the influence of surface tension forces which strive to minimize the surface area of the meniscus. Viscous losses which are caused by the motions of the fluid behind the meniscus cause the seesaw oscillation of the meniscus to decay with time and eventually halt.

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As such the time response of a nozzle during refill can be approximated by a damped second-order harmonic oscillator in which the mass of fluid entrained within the nozzle, firing chamber and refill port "bounces" on the compliance of the meniscus while viscous dissipation gradually damps out the oscillation. (It will be noted that none of the parameters involved - mass, compliance or resistance - are constants in this system; this is a linear approximation.)

During the brief time that the meniscus has overshot its equilibrium position and is bulging out of the nozzle, it is possible for the fluid in the bulge to spill out onto the material surrounding the lip of the nozzle. This spillage becomes very likely if the angle defined by the tangent to the meniscus bulge at the lip of the orifice equals or exceeds the wetting angle criterion for the material from which the nozzle plate has been manufactured. If this happens, the meniscus will break free from the Dip of the nozzle and the fluid bulge

will then spread out across the nozzle plate. As the meniscus retracts back into the bore, it reattaches itself to the edge of the nozzle and in so doing pulls most but not all of the fluid back down the bore with it. A small and very shallow puddle of ink is typically "stranded" in the immediate vicinity of the nozzle after each firing and refill cycle. At low frequency operating conditions - typically less than about 1,500 Hz -

5 ample time exists between firings for essentially all of this stranded ink to be wicked back up by the nozzles. However, at high frequency operation -typically greater than about 1,500 Hz and above - a new accumulation of puddled ink occurs at the nozzle lip. This accumulation can also occur at low frequencies if (1) the surface tension of the ink is sufficiently low, (2) the exterior surface of the orifice plate is sufficiently wettable, or (3) the back pressure (defined as the abso lute value of the static negative gauge pressure in the ink plenum) is sufficiently high (at least about -6 inches H₂O).

This accumulated ink has a deleterious effect upon print quality by capturing and deflecting the ejected ink droplet during the phase of droplet ejection when the tail is about to detach itself from the meniscus and follow the head of the droplet away from the nozzle. This causes breakoff to occur not from the retracted meniscus but instead from a random point around the wetted periphery of the nozzle; the drop is pulled off-

- 15 axis in the direction of the puddle. This direction error is integrated over the flight time of the droplet to result in a dot placement position error on the print medium. Since these errors are random in magnitude and direction, the result is an unpredictable and serious degradation of print quality. In some cases, the ink accumulation is severe enough to completely block droplet ejection from the nozzle.
- Hence, any providently designed ink jet printhead must include some features to minimize meniscus overshoot and minimize the time required for the meniscus oscillations to decay away, so that the preceding scenario (referred to as "nozzle wet-out") is avoided. It should be noted that wet-out can be caused or exacerbated by spray that breaks off the tail of the drop and rains back down on the nozzle plate. This is worst for low viscosity, high velocity drops.

Traditionally, wet-out is prevented by maintaining a static negative pressure, also known as back pressure, throughout the ink supply system so as to define an equilibrium position for the meniscus which lies inside the nozzle bore. Another method involves the use of anti-wetting coatings applied to the area surrounding the nozzle lip, which prevent meniscus breakoff during over-shoot. Yet another method is to increase the amount of viscous damping present in the ink supply system, thereby holding overshoot below the value required to initiate wet-out. Still another method is to provide a contact-line barrier that prevents the puddle from advancing out past a certain radius.

Anti-wetting coatings are of limited utility in preventing wet-out during overshoot since their lifetimes are typically shorter than that of the printheads to which they have been applied, causing wet-out to reappear prior to the completion of the printhead's service life. Furthermore, it is difficult to sufficiently immobilize these coatings so that wiping detritus from nozzles does not force the coating down into the bores, wreaking havoc irreversibly upon the printhead.

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It is often impossible in practice to draw down overshoot via static negative backpressure, since this backpressure acts to retard the refill of ink in the nozzles between firings. Hence, sufficient backpressure to prevent wet-out also compromises operating speed.

But a third option, increasing the amount of viscous damping has been found to be the most practical solution to the wet-out problem. This is because it (1) lasts the life of the pen, (2) does not slow refill so much that the firing frequency limit is compromised and (3) damps ripples and waves on the meniscus surface and hence makes the process more stable.

Increasing the hydraulic resistance (via ink-channel dimensional changes) is not the most practical method of increasing damping, at least in some situations, as printhead geometries push the limit of the smallest dimensions attainable with a particular material and process. (There is an ongoing need to scale down printhead geometries to allow the firing of smaller droplets, as is desirable when printing very high quality text, high resolution graphics or images containing gray levels or "halftoning". At drop volumes below 50 picoliters, nozzle wet-out due to insufficient damping becomes the dominant factor in degrading print quality.) The feed channel dimensions of such structures are already so minute that to include pinch

- points (as lumped resistive elements) in the feed channel structure would exceed the aspect ratio limits of the resist film from which the barrier structure containing the feed channels is formed. For Dupont "Vacrel" film, this aspect ratio is approximately 1:1. Hence, in Vacrel, a feature desired to be free of Vacrel must be at least 0.001 inch wide if the basis thickness of the film is 0.001 inch, 0.002 inch wide for 0.002 inch thick film and so on. Hence, to be manufacturable, some other means of obtaining sufficient meniscus damping to prevent wet-out must be included in the printhead structure.
 - Previous approaches to the problem of cross-talk, or minimizing inter-nozzle coupling, can be separated into three classes: resistive, capacitive, and inertial. The following is a brief discussion of each method and a critique of the typical embodiments of these methods.

Resistive decoupling (to hydraulically "decouple" the nozzles from one another) uses the fluid friction present in the ink feed channels as a means of dissipating the energy content of the cross-talk surges, thereby preventing the dynamics of any single meniscus from being strongly felt by its nearest neighbors. In the prior art, this is typically implemented by making the ink feed channels longer or smaller in cross-

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section than the main supply plenum. While these are simple solutions, they have several drawbacks. First, such solutions rely upon fluid motion to generate the pressure drops associated with the energy dissipation; as such, they can only attenuate the cross-talk surges, not completely block them. Thus, some cross-talk "leakages" will always be present. Second, any attempt to shut off cross-talk completely by these methods will necessarily restrict the refill rate of the nozzles, thereby compromising the maximum rate at which the printhead can print. Third, the resistive decoupling techniques as practiced in the prior art add to the inertia of the fluid refill channel, which has serious implications for the printhead performance (as will be explained at the end of the inertial decoupling exposition which follows shortly).

In capacitive decoupling, an extra hole is put in the nozzle plate above that point where the ink feed channel meets the ink supply plenum. Any pressure surges in the ink feed channel are transformed into displacements of the meniscus present in the extra hole (or "dummy nozzle"). In this way, the hole acts as an isolator for brief pressure pulses but does not interfere with refill flow. The location, size and shape of the isolator hole must be carefully chosen to derive the required degree of decoupling without allowing the hole to eject droplets of ink as if it were a nozzle. This method is extremely effective in preventing cross-talk (but can introduce problems with nozzle meniscus dynamics, as will be discussed below).

In inertial decoupling, the feed channels are made as long and slender as possible, thereby maximizing the inertial aspect of the fluid entrained within them. The inertia of the fluid "clamps" its ability to respond to cross-talk surges in proportion to the suddenness of the surge and thereby inhibits the transmission of cross-talk pulses into or out of the ink feed channel. While this decoupling scheme is used in the prior art, it requires considerable area within the print head to implement, making a compact structure impossible.

25 Furthermore, since the resistive component of a pipe having a rectangular cross-section scales directly with length and inversely with the third power of the smaller of the two cross-section dimensions, the flow resistance can grow to an unacceptable level, compromising refill speed. More importantly, however, are the dynamic effects caused by the coupling of this inertance to the compliance of the nozzle meniscus, as will be discussed below.

With regard to the problem of meniscus dynamics, there are apparently no solutions offered in the prior art. Apparently, this is a problem that has only recently surfaced as printhead designs have been pushed to accommodate higher and higher repetition rates. Clearly, any method used to decouple the dynamics of neighboring noz zles will also aid in damping out meniscus oscillations, at least from a superficial consideration. In practice, problems are experienced when trying to use the decoupling means as the oscillatory damping means. These problems can be traced to the synergistic effects between the nozzle meniscus and the fluid entrained within the ink feed channel, as outlined below.

If resistive decoupling is attempted by reducing the width of the entire ink feed channel, the inertia of the fluid entrained within the feed channel increases. When this inertia is coupled to the compliance of the meniscus in the nozzle, it results in a lower resonant frequency of oscillation of the meniscus, which requires a longer settling time between firings of the nozzle. The inertial effect and the resistive effect are tied together, with the net effect being that settling time cannot be reduced.

Capacitive decoupling has been proven effective at droplet ejection frequencies below that corresponding to the resonant frequency of the nozzle meniscus coupled to the feed channel inertia. However, its implementation at frequencies near meniscus resonance is also complicated by interactive effects. Specifi-

- 45 cally, the isolator orifice acts as a low impedance shunt path for high frequency surges. Hence, the high frequency impedance of an ink feed channel terminated at its plenum end with an isolator orifice will be lower than an equivalent channel without an isolator. This means that during the bubble growth phase, blow-back flow away from the nozzle is increased by the isolator orifice. This robs kinetic energy from the droplet emerging from the nozzle, which results in smaller droplet size and lower droplet velocities and thus lower
- 50 ejection efficiency. During the bubble collapse phase, the isolator orifice meniscus pumps fluid flow back into the refill chamber, which excites a resonant mode in which the two menisci trade fluid between themselves via the ink feed channel. Since these two menisci are for most practical designs similar in size, and since they are effectively "in series", the equivalent compliance of the coupled system is roughly half of that with only one orifice in it. The two-orifice system will thus resonate at a higher frequency, which is a
- 55 benefit from a settling time point of view, but the energy stored in the resonating system still needs to be dissipated and therefore constrictive damping will be necessary in such an implementation. While the effects of these resonances is poorly understood at this time, the efficiency decrease may be severe enough to prevent the printhead from working.

It is clear that what is needed is a method of printing that accomplishes both (1) isolation of any given nozzle from its neighbors and (2) reduced oscillation of the meniscus during refill (to minimize interference with the ejection of subsequently fired droplets. This method must do the above while not introducing any adverse side effects.

DISCLOSURE OF THE INVENTION

In accordance with the present invention, the viscosity of the jetted fluid is adjusted to control the quantity of damping present in the fluid supply channels or refill ports of the ink jet printhead. Since any viscosity increase acts to increase viscous damping present throughout the ink supply circuit, the feed channel dimensions in the supply circuit may be increased in order to prevent excessive pressure drops within the supply circuit. From a processing and manufacturing standpoint, enlargement of these features is simple, in contrast to the much more difficult problem of making the same features smaller, as would be required to enhance damping via the traditional techniques discussed above in the Background Art.

There are two examples of how this principle may be used to enhance the operation of ink-jet printheads. In the first example, the directionality problem arising from nozzle wet-out, referred to as "streaking", is eliminated via an ink viscosity increase from an original value of 5 cp to an adjusted value of 7.5 cp.

20 7.5 cp.

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In the second example, the issue of insufficient damping at the manufacturable limit of the barrier structure is addressed. In this case, the channel architectures are enlarged to accommodate the thicker ink. The original ink had a viscosity of 1.2 cp. The intermediate ink viscosity was 5 cp. The thickest ink had a viscosity of 11 cp.

This invention involves adjustment of ink viscosity as a means of enhancing printhead performance in situations where hydraulic tuning is impossible or impractical, i.e., head architectures which are already at the limits of manufacturability and/or which are no longer available for changes due to other design constraints. Adjustment of ink viscosity allows an otherwise impossible range of tradeoffs between nozzle refill and meniscus settling time to be made in such printheads. The operating speed improvements which this technique permit are quite large: a three- to five-fold increase in operating speed over current state of the art.

While the drop stability in the pen is improved with an increase in the viscosity of the ink, such increase in viscosity does not generate crusting, clogging and problems in print quality over the entire environmental operating range (typically given as 3° C/70% RH to 15° C/20% RH, where RH is relative humidity).

This invention allows small drop-volume printers to operate without the ordinarily-encountered stability problems at droplet ejection rated above 10 kHz. It also allows the dynamics of ink droplet formation to be decoupled from the wettability of the orifice plate, which prevents uneven frequency response, trajectory errors and air ingestion. It allows these printheads to avoid such characteristics even in those situations where similar tuning efforts (via inclusion of lumped resistive elements, for instance) are prohibited.

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BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a perspective view of a nozzle plate and nozzles therein, depicting an emerging droplet of ink from a nozzle and a puddle of ink associated therewith;

FIG. 2 is a cross-section taken along the line 2-2 of FIG. 1, showing the structure of one particular drop generator; and

FIG. 3 is a view similar to that of FIG. 2, showing a portion of an ink-filled drop generator with a bulging meniscus and a drop of ink wetting the nozzle plate at characteristic wetting angle θ_w .

BEST MODES FOR CARRYING OUT THE INVENTION

Referring now to the drawings wherein like numerals of reference designate like elements throughout, a portion of a printhead is depicted in FIG. 1. In particular is seen a nozzle plate 10 in which are recessed a plurality of nozzles 12 in individual recesses 13. Ink 14 is fired from resistors through the nozzles in a particular arrangement toward a print medium (e.g., paper) to form alphanumeric characters and graphics. FIG. 2 depicts a portion of a feed chamber 16 in which is located a resistor 18; there is one resistor associated with each nozzle 12. Ink is fed into the feed chambers from a plenum (not shown). Upon receiving a pulse of energy from an external source, the resistor 18 is heated to a level sufficient to expel a droplet of ink 14 toward the print medium. Following ejection of the ink droplet 14, additional ink fills the chamber 16 in preparation for another firing.

The nozzle 12 has a nozzle diameter d; each resistor covers a square area with side dimension s; the channel width is given by w. The thickness of the nozzle plate 10 is t_p , while the thickness of barrier layer 20 is t_b . In a preferred example, the printhead employs a barrier layer 20 comprising Vacrel 55 μ m thick and a nozzle plate 10 comprising gold-plated nickel 63 μ m thick. The nozzles 12 are 47 ±3 μ m diameter, with resistors 64 μ m x 64 μ m, and channel width 84 μ m wide.

As indicated in the Background Art section, during the overshoot phase, a puddle 22 of ink may form adjacent the nozzle 12. If not wicked back into the chamber, such a puddle may have a deleterious effect upon print quality by interfering with the droplet 14 of ink as it is ejected from the nozzle 12.

During the refill process, the meniscus overshoots its equilibrium position, is slowed, stopped, and eventually reversed by the surface tension of the meniscus. The maximum overshoot occurs when the meniscus is stopped. In FIG. 3, θ corresponds to the maximum overshoot of the meniscus. The angle θ is defined by a tangent to the meniscus surface at the nozzle perimeter and a line drawn parallel to the top plate surface. To avoid spillage onto the top plate, θ should be less than θ_w , the characteristic wetting angle for the ink and top plate materials.

As used herein, a stable drop generator is one that makes drops with consistent trajectories, volumes, speeds, and break-up patterns. In accordance with the invention, this stability becomes more likely as the viscosity is increased. This is because it is the damping effect of viscosity that will balance and control the inertial and surface forces that drive the refill and ejection processes. Unstable drop generators with low viscosity are characterized by chaotic meniscus movement, large meniscus overshoots, erratic spray patterns, and puddles 22.

This stability can be measured by looking at the accuracy and consistency of dot placement and size. Stability was measured by looking at line spacing on paper. The odd-numbered nozzles in the pen were fired across the page, forming a set of parallel lines. Then, an identical pattern was made with the evennumbered nozzles on a different part of the page. A vision system then examined the patterns, measuring line spacing uniformity and line width uniformity.

These measurements of line spacing and width were then combined into an overall "print quality number", with 4 being a perfect grade. Testing at 30 $^{\circ}$ C (the worst-case operating temperature for print quality) revealed that 40% H₂O/60% DEG ink (DEG is diethylene glycol) had a print quality number of 3.2, which was a full two points better than the less viscous 50% H₂O/50% DEG (print quality number of 1.2).

35 Also, considering dot placement only, measurements of cross-scan directionality using the same plot showed that going from an ink viscosity of 5 to 7.5 cp (50/50 water/DEG to 40/60 water/DEG) decreased the variation in angular misdirection by 43%:

TABLE I.

Printing Results (30° C/70%RH)			
lnk	ambient viscosity	cross-scan 3-sigma	
(%H ₂ O/DEG)	(cp)	(degrees)	
50/50 40/60	5.0 7.5	1.20 0.69	

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Computer modeling of the ink flow in the printhead confirmed the improved stability obtained with the higher viscosity ink modeling of a pen employing 59 μ m Vacrel and nozzles 12 having a diameter of 43 μ m and showed that a change in vehicle composition from 50/50 water/DEC to 40/60 water/DEC should result in the following changes in refill time, overshoot, and damping:

TΑ	BL	_Е	11.

% Change Relative to 50/50 at 30° C				
lnk	Temp.	Refill	Overshoot	Damping
40/60 H ₂ O/DEG 40/60 H ₂ O/DEG	60°C 30°C	+ 2.3% + 18.2	-26.1% -49.3	+ 36.9% + 67.4

In another experimental comparison, 50/50 ink evidenced a spear drop onset at 3,500 Hz. (Spear drops are headless, very fast, and usually misdirected; they appear above certain critical frequencies.) 40/60 ink evidenced a spear drop onset at frequencies of about 4,800 Hz, while 30/70 ink evidenced a spear drop onset at frequencies greater than 5,500 Hz.

15 In yet another experimental comparison, various compositions of ink were fired from pens with the following results:

TABLE III.

Properties for Various Viscosities of H ₂ O/DEG			
% H ₂ O	V _{min} /V _{ss}	Directionality	Viscosity cp (25 [°] C)
60	0.729	1.5	3.89
50	0.833	4	5.48
40	0.860	7.5	8.61
30	>0.95	9	13.81
20% NMP	0.901	7.5	7.94
Notes: (1) V_{min}/V_{ss} is the ratio between the minimum velocity in a frequency resonance plot and the steady state velocity. A value close to 1.0 indicates stability over the freque cy range. (2) Directionality is on a scale of 0 to 9, where 0 is bad and 9 is good. (3) 20% NMP = 40% H ₂ O, 40% DEG, 20% N-methyl pyrrolidone.			

Print quality was determined for a variety of compositions, using the preferred printhead configuration given above. The results are listed below, with average print quality given for the indicated vehicle composition. The higher the value, the better the print quality. Each pen has three groups of ten nozzles each; each such group is called a primitive. In the test, a visual determination was made for each half-primitive (the odd or even nozzles), and summed for all six half-primitives to arrive at the average PQ rating. The rating is based on 0 = very poor, 1 = poor, 2 = fair, and 3 = good; values of 2 and above (here, 12.0)

and above) are deemed acceptable.

TΑ	BL	ΕI	V.

Print Quality vs. % H ₂ O/DEG		
H₂O/DEG	Ave. PQ Rating	
70/30	0.8	
60/40	8.0	
50/50	11.9	
40/60	16.5	
30/70	18.0	
5% NMP	14.0	
10% NMP	13.8	
15% NMP	16.0	
Note: % NMP plus equal portions of H ₂ O		
and DEG		

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From the foregoing, it is evident that an increase in viscosity of ink improves the print quality considerably. However, there is an upper limit on the viscosity of ink that may be employed, since higher viscosity inks take longer to dry and increase the refill time. Indeed, in conjunction with some print media (e.g., Mylar transparencies), the upper limit is severely constrained. For example, 30/70 H₂O/DEG is useful with paper, but cannot be used with Mylar transparencies.

Although diethylene glycol was used to increase the viscosity of the inks in the foregoing examples, it will be readily clear to those skilled in this art that the teachings of this invention are applicable to any of the water-miscible glycols typically used in ink-jet printing. Thus, in addition to diethylene glycol, ethylene glycol and propylene glycol are but a few examples of the many glycols that are used in ink-jet printing, and an increase in the glycol content relative to water will accomplish the same purpose, with the same end result as indicated above.

35 Claims

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1. A method of increasing drop stability in a pen employed in an ink-jet printer having a plurality of nozzles (12) in a nozzle plate (10) associated with a drop-firing means (18) for firing drops of ink (14) through said nozzles, **characterized** in that the viscosity of said ink is increased.

2. The method of claim 1, characterized in that the glycol content relative to the water content is increased.

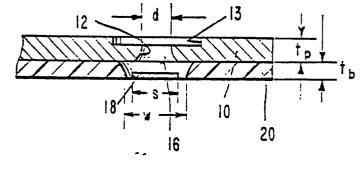
3. The method of claim 1 or 2, characterized in that the viscosity is increased by at least about 50 %.

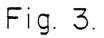
4. The method of claim 3, **characterized** in that said ink comprises a vehicle consisting essentially of about 30 to 40 % water and the balance diethylene glycol.

5. The method of claim 4, **characterized** in that said ink comprises a vehicle consisting essentially of about 40 % water and about 60 % diethylene glycol.

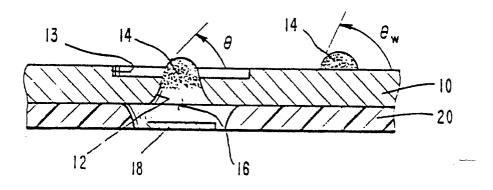
6. A pen of an ink jet printer for performing a method according to one of the preceeding claims, **characterized** by a plurality of ink feed chambers (16) defined in a barrier layer (20) comprising Vacrel about 55 μ m thick and a nozzle plate (10) comprising gold-plated electro-formed nickel about 63 μ m thick,

with a plurality of nozzles (12) formed in said nozzle plate about 47 μ m diameter, and with a resistor (18) about 64 μ m x 64 μ m formed in the bottom of each of said chambers and operatively associated with one of said nozzles, said ink feed chambers fluidically communicating with a source of ink (14) by an ink feed channel having a width 90 μ m wide at the top thereof and wherein said viscosity of ink is at least about 7.5 cp at 30° C.

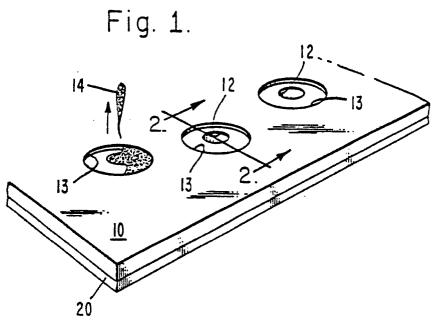




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